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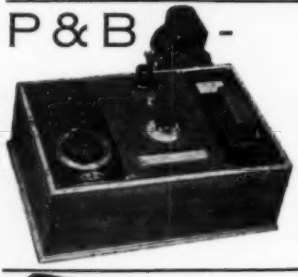

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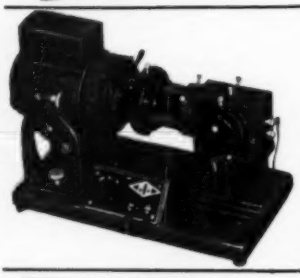

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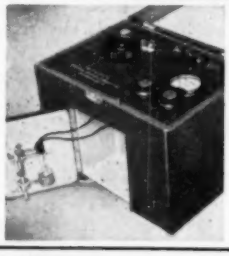

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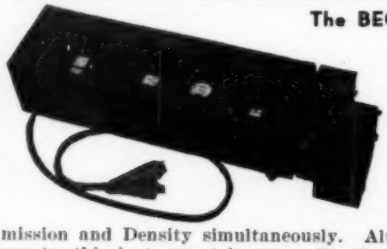

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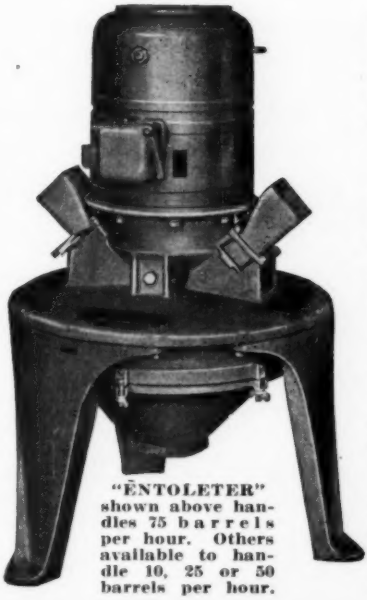

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CEREAL CHEMISTRY

VOL. XX MAY, 1943 No. 3

THE PENTOSANS OF WHEAT FLOUR

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Wallace & Tiernan Laboratories, Newark, New Jersey

(Read at the Annual Meeting, May 1942)

Baker and Mize (1942) reported that sodium chlorite was decomposed in 24 hours by the solution obtained in washing gluten from a dough. Chlorite was also reduced by glutens that were dispersed and suspended in salt solutions. Little or no sodium chlorite was reduced under similar conditions by either the starch or the so-called "amylodextrin" portions separated from the dough by the washing. It was also shown that the flow property of gluten could be largely removed by thorough washing of the gluten, indicating that this "flow" property was associated with the soluble constituents of dough.

In studies reported above on the effect of oxidation with sodium chlorite on the elastic spring and viscous flow of doughs, it was found that the flow was greatly decreased in the dough by a moderate degree of oxidation, and that a high degree of oxidation of the yeastless dough caused tightening of the dough and a marked decrease in flow. These observations led to the subsequent study of the water-soluble material in flour and the effect thereon of several oxidizing agents.

Experimental

Preparation of strong gluten wash water: Gluten was washed from a dough in an equal weight of 3½% salt solution (instead of ¾% salt solution as described in work referred to above). The starches were separated from this solution by centrifuging and the supernatant liquid was used again and again to continue the rewashings of the gluten until the starch was as completely removed as possible.

Properties of strong gluten wash water: When to 5 ml of such strong gluten wash water one or two drops of 0.1% solution of sodium chlorite were added, and allowed to stand a short period, a gel was formed.[1]

[1] Five milliliters represented extract from approximately 2½ g of flour. One drop of 0.1% sodium chlorite was approximately 20 ppm, basis of 2½ g of flour.

This gel resembled undisturbed egg white and had many of its properties. It could be whipped to a stiff foam resembling beaten egg white. This unbeaten gel became a liquid when allowed to stand over-night at room temperature. It would not re-gel on further standing, nor could it be induced to gel by the addition of more sodium chlorite.

Preparation of Concentrated Flour Extract

The above method of preparing concentrated solutions of flour solubles was so laborious that a simpler method was devised.

One hundred grams of flour was beaten with 200 ml of distilled water for five minutes in a Waring Blendor. Oxygen and foam were eliminated from the operation by placing the Waring Blendor in a vacuum chamber. The batter thus obtained was poured into centrifuge tubes covered with cellophane and whirled in an ordinary Babcock centrifuge for 30 minutes. The supernatant liquids thus separated are the concentrated flour extracts referred to in this paper, and were so prepared unless otherwise indicated.

Properties of Concentrated Flour Extracts

These concentrated flour extracts reacted toward sodium chlorite as did the strong gluten wash water. Gels were formed at the 20- to 40-ppm concentration of sodium chlorite, but would not form when 80 ppm was added.

These gels also became liquid when allowed to stand overnight at room temperature or when boiled for a short time. Lowering the temperature of the gels below 17.5°C delayed liquefaction. Once the gels were liquefied they could not be reconstituted, even by the addition of more sodium chlorite.

Hydrogen peroxide, sodium iodate, iodine in potassium iodide, potassium bromate, sodium meta-vanadate, ammonium persulfate, oxygen, and calcium peroxide (all commonly used and well known oxidizing agents) gave, to some degree at least, gels that were similar to those obtained with sodium chlorite. When potassium bromate was used, it was necessary to saturate the concentrated flour extract with carbon dioxide to form a gel.

Separation of the Gel-Forming Substance

Since there was apparently something in these flour extracts, as well as in the strong gluten wash waters, that could be caused to stiffen or gel by the addition of an oxidizing agent, the separation of this ingredient was attempted. At first it seemed that the substance was a soluble protein, and so various protein precipitants were tried. Most

of the precipitates obtained by the use of various alcohols, acetone, and salts would not gel with an oxidizing agent when redissolved.

The addition of ammonium sulfate and certain dilutions of ethyl alcohol gave precipitates which, upon redissolving and dialyzing, gelled with either sodium chlorite or iodine. Such precipitates contained less than 50% protein, thereby giving the first indication that the material which gelled might have been nonprotein. Solutions of these dialyzed ammonium sulfate precipitates, when hydrolyzed with acid and then neutralized, could not be fermented with yeast yet such hydrolysates had strong reducing properties with Fehling's solution.

Guided by these observations, pentoses were tested for and found in abundance. This indicated that very possibly pentosans were the water-soluble gelling ingredient. Pentosans have been reported in flour by other workers. Jacobs and Rask (1920) worked out a laboratory method for the control of wheat flour milling based upon the determinations of pentosans. Hoffman and Gortner (1927) reported separating soluble pentosans from some of the residues obtained when carrying out Osborne's classical method of preparing soluble proteins. Their method was not suited to our purpose because of the long periods of time through which the solutions must be retained before the pentosans could be separated. Larmour (1927) described a pentose containing material obtained by alkaline extraction of cereals. His method was not adaptable because the alkaline extracts would not gel.

Durham (1925) in his work on viscosity and hydrogen peroxide in flour suspensions reported the formation of a gel from a solution similar to our concentrated flour extract. He was unable to separate any constituent from the gel.

In efforts to develop quicker and more direct methods of separating this substance, concentrated flour extract was fractioned with increasing amounts of ammonium sulfate; the steps included separating, dialyzing, redissolving, then repeating. No product was obtained that contained less than 20% protein, although nearly all of these fractions gave gelling reactions with sodium chlorite.

Finally, after many trials and modifications of Ritthausen's method (1872), clear filtrates were obtained that were nearly protein-free, and by a second treatment with the copper reagent, followed by precipitation with ammonium sulfate, pentosan products were obtained which contained as little as 0.1% protein.

Method of Preparing Pure Water-Soluble Wheat Gum

Four hundred grams of flour was mixed with 800 ml of distilled water in a 2-liter flask by shaking together, and then further mixed in a vacuum with a Waring Blendor operated at top speed for 5 minutes.

(This required two batches.) The batter was poured into centrifuge tubes, covered with cellophane held by rubber bands, and centrifuged for 30 minutes.

The supernatants were filtered through a cotton-plugged funnel. To every 100 ml was added 16.7 ml of saturated $CuSO_4$ at 25°C. The solution was then stirred with a high-speed stirrer with a hexagonal nut for an agitator. During stirring 6.0 ml of 5 *N* NaOH was added gradually. A burette having a capillary jet was used so as to direct the NaOH just clear of and between the parallel planes of the stirrer head.

Perceptible particles of cupric hydroxide sometimes formed in the suspension. Continued mixing disintegrated these and a homogeneous mixture was obtained. The pH of the mixed solution was between 5.5 and 5.6 at this point. Usually the precipitate settled rather sharply and rapidly.

This solution was transferred to metal cups protected by thin coatings of bees wax, and centrifuged at high speed, 5000 ppm for 30 minutes. The centrifugates were collected by filtering through a cotton plug. The filtrate was usually clear, though with some flours there was slight turbidity.

A second copper precipitation was carried out upon this filtrate precisely as had been done above. The pH of the mixed solution, after the addition of the caustic soda solution, was adjusted if necessary to between 5.8 and 6.0.[2] This solution was also centrifuged and filtered. Usually this filtrate was brilliantly clear and only slightly colored with copper sulfate.

Having removed the soluble proteins, our next step was to precipitate the pentosan gum with ammonium sulfate. To every 100 ml of filtrate obtained above was added 70 grams of cp ammonium sulfate. This was dissolved as much as possible by stirring. The gum thus precipitated was allowed to rise to the surface and pack together. This gum, along with the supernatant liquid, was decanted from the undissolved ammonium sulfate into centrifuge cups. After centrifuging for 15 minutes at moderate speed, the gum forms a tough floating layer. This was removed and rinsed rapidly with a very little distilled water. It was then dissolved in distilled water by mixing with water a little at a time. A clear homogeneous solution was slowly obtained. The gum solution was made up to approximately 100 ml and dialyzed in a collodion bag. The ammonium sulfate was nearly all removed in 24 hours by dialysis at 15°–17°C against 2000 ml distilled water renewed 5 times.

[2] The pH was determined by a colorimetric comparator method with methyl red as the indicator and a phosphate buffer. A clear water background was used for the unknown sample containing the indicator and the unknown solution itself (with its copper color) was used as a background for the buffer solution containing the indicator.

To obtain the purest gums, the dialyses were carried on for 48 hours. It was noted that these pure gums would tend to gel in the bags if the dialyses were continued for a longer time. This seemed to be due to oxygen dissolved in the distilled water. Many of the purer gums were dialyzed against cold boiled distilled water that had been saturated with CO_2.

The pure gum may be precipitated from the dialyzed solution by 70% alcohol, or by removal of water by vacuum at room temperature. Most of our studies were carried out with the purified dialyzed solutions.

The analyses of these water-soluble wheat gums obtained as above showed them to be substantially pure pentosans. The furfural method (*Cereal Laboratory Methods*, 4th ed. 1941, pp 68–69) accounted for approximately 95% of the compound. No uronic acid and no galactose could be found. Its composition was found to be similar to that reported for flour pentosans by Freeman and Gortner (1932).

Properties of Soluble Wheat Pentosan

These purified wheat pentosans dissolved slowly in water to clear solutions which were highly viscous as 1% concentrations. The solutions would not whip to a foam as did the concentrated flour extracts. They did not gel as readily with all oxidizing agents as did the concentrated flour extracts. Best gels were obtained when iodine (in KI) was added in barely detectable excess, using starch indicator. Sodium chlorite also gave a gelling reaction. Sunlight and ultraviolet light both hastened the gelling reaction of oxidizing agents on solutions of pentosans, as well as on concentrated flour extracts.

Table I gives data obtained from a series of flours of widely varying characteristics. The total soluble pentosans found in 10 ml of concentrated flour extract, the amount of purified pentosans recovered from 10 ml of the flour extracts, and the percentages of nitrogen found in the purified pentosans are tabulated. The last two columns show the gelling reactions for both the flour extracts and the purified gums.

The amounts of total soluble pentosans found in the concentrated flour extracts did not vary widely except for Chiefkan flour, which contained the least. The amounts of purified pentosans recovered were not especially significant because of the influence thereon of the technique used in their purifications. In some cases yields of 45% were obtained. The pentosans not recovered were largely lost in the copper precipitate. Precipitation by ammonium sulfate is not complete and hence more pentosans are lost in this step.

The data indicating the gel reactions of the flour extracts and of the gums purified therefrom show that some of the flour extracts did not gel by any of the methods used. Two of them did gel when a minimum

of water was used in making the concentrated flour extracts. The purified pentosans from extracts of all sound flours possessed the gelling property. Those from sprouted wheat flours did not gel. The reasons for the failure of some of the flour extracts to gel with oxidation was not indicated by the experiments recorded in Table I. One might expect

TABLE I

SOME CHARACTERISTICS OF THE SOLUBLE PENTOSANS FOUND IN SEVERAL FLOURS

Kind of wheat flour	Pentosans in 10 ml of flour extract	Purified pentosans recovered from 10 ml of flour extract	Nitrogen found in purified pentosans	Gel reactions	
				Flour extract	Purified pentosans
	g	g	%		
Kansas hard winter patent	0.060	0.024	0.10	Gel	Gel
Dakota hard spring patent	0.064	0.014	0.02	Gel	Gel
Dakota hard spring clear	0.049	0.018	0.20	No gel	Gel
Kansas chiefkan (1939 crop)	0.038	0.020	0.21	No gel [1]	Gel
Dakota durum	0.057	0.023	0.22	No gel	Gel
Eastern soft winter	0.050	0.023	0.24	No gel [1]	Gel
Minnesota, 27% sprouted	0.061	0.011	0.38	No gel	No gel
Minnesota, 10% sprouted	0.062	0.025	0.38	No gel	No gel

[1] Gelled when extracted with less water.

all sound flour extracts to gel because their corresponding gums purified therefrom did gel. Possibly there were some other flour ingredients in those concentrated flour extracts that interfered with the gel formation.

Effect of Various Substances upon the Viscosity and Gelling Property of Purified Pentosans

The effects of many substances upon the gelling property of purified wheat pentosans were tested to determine whether any of them had this interfering characteristic. The following substances did not prevent the gelling reaction: maltose, soluble starch, peptones, papain, pancreatin, and purified alpha-amylase. Egg white and milk had the effect of retarding the formation of a gel, and the gels thus formed were softer than otherwise.

Interferences with the gelling reaction were obtained by the use of heated malt extract, unheated malt extract, water extract from sprouted wheat flour, water extract from wheat bran, and water extract from wheat germ.

In the case of bran extract, a gel could be formed if sodium chlorite was added promptly after the bran extract had been mixed with the

solution of pentosans. But such gel would liquefy very soon after its formation. If the bran extract was left in contact with the pentosan solution for half an hour before the oxidizing agent was added, no gel could be formed. These experiments suggested enzymatic action and so tests were applied for reducing sugars. Since these tests were negative, it indicated that the enzymatic action had not proceeded far enough to produce pentoses yet might have reduced the size of the aggregates or otherwise altered the pentosans.

Effect of Concentration on Viscosity and Gelling Property of Soluble Pentosans

The data given in Table I show that some of the flour extracts gave a gel reaction only when extracted with less than the usual amount of water. This led to a study of the viscosities of flour extracts and solu-

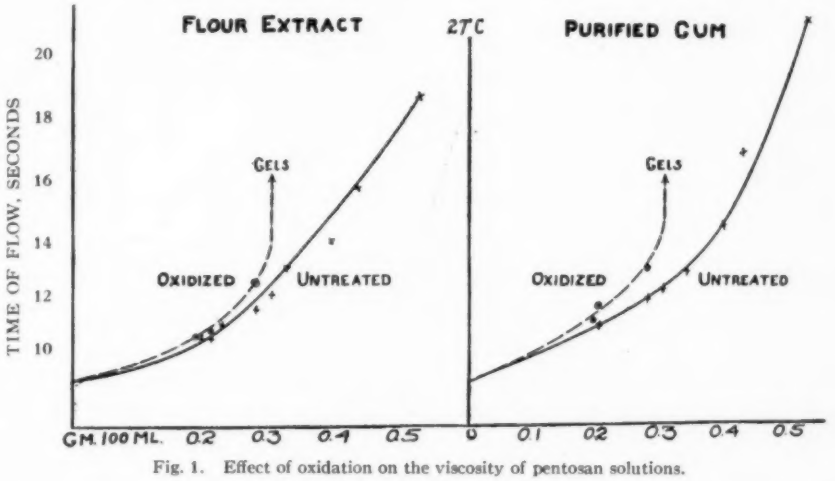

Fig. 1. Effect of oxidation on the viscosity of pentosan solutions.

tions of purified wheat pentosans before and after the addition of oxidizing agents. The data obtained from these experiments are given in Figure 1.

Two sets of curves are shown. That on the left gives the results for flour extracts and that on the right for purified gums (wheat pentosans). The ordinates show the seconds of flow from a pipette and the abscissas show the grams of pentosans per 100 ml for each of the solutions tested.

The viscosities of the untreated (unoxidized) solutions rose regularly with increased concentrations. When oxidizing agents were added, similar increases in viscosities were observed until the critical concentrations of the solutions were reached. At these points the viscosities quickly rose to immeasurable values because the solutions turned to gels. It is to be noted in passing that the two sets of curves,

the ones for flour extracts and the ones for purified gums, are quite similar.

Effect of Temperature and Concentrations on the Viscosity of Pentosan Solutions

Baker and Mize (1939) suggested that "The changes in dough which produce undesirable results are those associated with excessive softening and loss of viscosity during heating." That observation led us to make determinations of the effects of varying temperatures upon

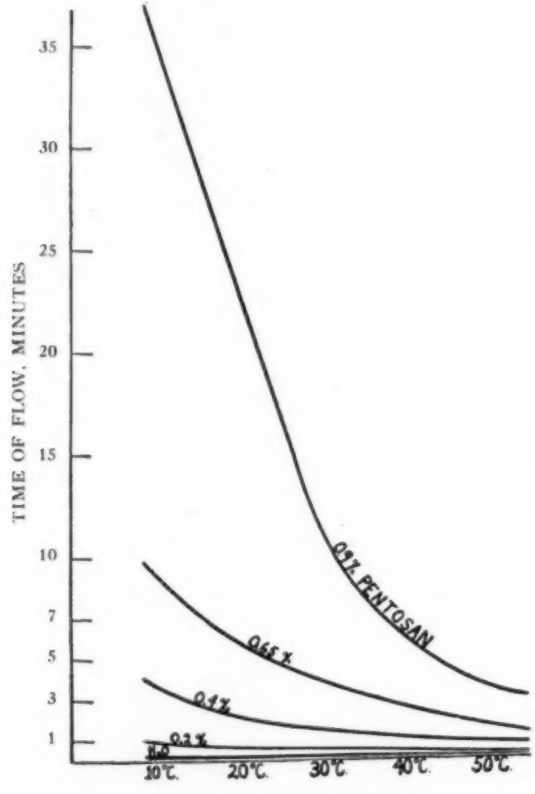

Fig. 2. The viscosity of purified pentosan solutions.

the viscosities of pentosan solutions. Figure 2 shows the results of these studies.

The ordinate scale shows viscosity at time of flow from a pipette and the abscissa the temperatures at different concentrations of purified pentosan solutions. The flow of water through the same pipette is shown by the lower curve for comparison. The solutions all showed relatively high viscosities which increased greatly as the concentration

was increased. Viscosities dropped rapidly when the solutions were heated from 8°C to 65°C. Though not shown in the figure, the addition of 50 ppm of glutathione to these pentosan solutions further lowers their viscosity appreciably.

Character of Pentosans Obtained from Oxidized Batters

It was observed during the progress of these studies that when a gel was once formed and subsequently broken up by stirring or beating, it could not be reformed either on standing or by further oxidation. In order to indicate whether this effect occurs in batter or dough, a series of experiments was undertaken where sodium chlorite was added to a concentrated flour batter 25 minutes before centrifuging in order to permit the batter to thicken.

TABLE II

EFFECTS OF SODIUM CHLORITE UPON THE WATER SOLUBLES OF A KANSAS HARD WINTER WHEAT PATENT FLOUR

Treatment of batter	Soluble pentosans in 10 ml of water extract	Soluble protein in 10 ml of water extract	Total soluble extract in 10 ml of water extract	Gel reactions	
				Flour extract	Purified pentosans
	g	g	g		
No treatment,	0.060	0.076	0.364	Gel [1]	Gel [2]
$NaClO_2$, 25 ppm	0.056	0.070	0.358	Gel [3]	Gel [3]
$NaClO_2$, 80 ppm	0.050	0.070	0.352	No gel	No gel

[1] Stiffest, best gel. [2] Medium gel properties. [3] Poorest gel reaction.

Table II shows the results of this study. Three batters from the same flour were made up—one with no oxidizing agent therein, the second with 25 ppm sodium chlorite added, and the third with 80 ppm of $NaClO_2$. The batters containing $NaClO_2$ thickened appreciably.

The soluble pentosans were determined in 10-ml portions of the concentrated flour extracts centrifuged from the three batters. Each figure in the first column represents the grams of soluble pentosans in 5.0 g of flour. The second column shows the grams of soluble protein matter and the third column the grams of total soluble matter in 5.0 g of flour. The gel reactions of the concentrated flour extracts and of the purified pentosans obtained therefrom are shown for each flour treatment in the last two columns.

It is to be noted that with increased oxidation the amount of soluble pentosans decreased nearly 20%. There was a somewhat similar decrease in the soluble proteins. The total soluble extract decreased by an amount about equal to the sum of the losses of those two ingredients in the supernatant. The gel reaction on oxidation was cor-

respondingly less firm as the oxidation in the batter was increased until, when 80 ppm of $NaClO_2$ was added to the batter, neither the concentrated flour extract nor the purified pentosans separated therefrom possessed any gelling property.

Insoluble Pentosans in Wheat Flour

Jacobs (1920) found that refined flours contained from 2.25% to 2.80% of total pentosans, on the dry-flour basis. We found part of the wheat-flour pentosans to be water-soluble. A thorough extraction of a flour with water by decantation indicated that the total soluble pentosans averaged above 1.0%. The insoluble pentosans must be present either in the gluten or the starch; otherwise an impossibly large percentage of bran powder must be present to account for such a relatively large amount of insoluble pentosans.

TABLE III

INSOLUBLE PENTOSANS IN WHEAT FLOUR

Constituent	Protein $N \times 5.7$	Pentosans redistilled	Average diameter of starch granules
	%	%	μ
Gluten, 1st washing	84.9	0.95	—
Gluten after 4th dispersion	94.4	0.66	—
Starch, large granules, rewashed 4 times	0.3	0.44	21.4
Starch, small granules, rewashed 4 times	2.2	14.01	3.9

In order to locate these insolubles, we separated the gluten and starch by ordinary gluten working technique with a relatively large amount of water. The gluten was washed four times by the dispersion method as described by Baker, Parker, and Mize (1942) while the starch was washed four times by decantation to remove all solubles.

The starch was then collected by centrifuging. It deposited in three layers in the centrifuge cup. The lower layer contained the large starch granules. A thin, sharply defined intermediate layer was mostly bran powder, while the upper or third layer was made up of small granules of starch, which have sometimes been called "amylodextrin." This upper layer was carefully removed from the cup; the intermediate layer was removed and discarded. The lower layer was washed and centrifuged and kept apart from the upper small-starch layer which also was further washed and centrifuged until finally two well washed and free-from-bran-powder samples of starch were obtained, one of coarse granules and the other of fine granules. These were analyzed for pentosans by the method of Schmidt, Neilson, and Hammer (1932). The washed and dispersed glutens were also an-

alyzed for pentosans. Table III gives the results of these determinations.

The hand-washed gluten (first washing) contained 0.95% pentosans. Four dispersions of this gluten only lowered this quantity to 0.66%—indicating that there was only a small amount of soluble pentosans removable from the gluten. The small residual may well have been due to the retention of bran particles in this hand-washed gluten. The rewashed (4 times) coarse starch contained 0.44% pentosans and 0.3% protein, while the small-granule starch contained 14% pentosans and 2.2% protein.

The high pentosan content of the small starch could not have been due to the presence of bran powder for two reasons—first, because it would have required a 60% bran content to have given such a value and, second, because a microscopic examination showed practically no bran particles among the spherical starch granules.

By analysis, there was 33 times as much water-insoluble pentosan in the small-starch as in the large-starch granules. Measurements of the average diameters of these starch granules showed that the larger granules averaged 21.4 μ, while the small ones had an average of only 3.9 μ. The ratio of the surfaces for equal weights of the small and large starch granules was calculated by the formula: $(D_1)^2/(D_2)^2 = 30.4$, where D_1 = diameter of large-starch granule and D_2 = diameter of small-starch granule.

The small starch had 30.4 times as much surface and 33 times as much pentosan as did the large starch. These similar values are good indications that all the starch granules, both large and small, have a pentosan content proportional to their surfaces and suggests that the pentosans are deposited upon the surface of the starch granules.

Discussion

There has been presented in a somewhat chronological order experiments that indicate the presence of soluble pentosans in wheat flour. These usually have the property of forming aqueous gels when treated with certain oxidizing agents.

There were many variations in the effects of the several commonly used oxidizing agents upon both the concentrated flour extracts and the purified pentosans separated from them. These variations were further complicated by the changes brought about by temperature, concentration, time, and reducing substances such as glutathione.

It is shown that several soluble ingredients found in flour and bread doughs interfered with this gelling reaction. For example, maltose, soluble peptones, papain, pancreatin, and alpha-amylase had little or no effect upon the gelling property of pentosans, while other constitu-

ents of doughs such as malt extracts, water extracts of sprouted wheat flour, bran, or germ all interfered seriously with the formation of gels.

These studies and observations lead to a consideration of the possible role that pentosans may play in the production of bread.

In sound patent bread flours there is some variation in the amount of soluble pentosans present. The soluble pentosans usually amount to 1% or more in refined flour. If all the soluble pentosans in dough are in solution in the free water of the dough and the free water is 50% of that added, as shown by Skovholt and Bailey (1935), there would be approximately a 4% solution of pentosan in the free water. Doubtless the actual concentration is somewhat lower, but in any case dough should contain a much more concentrated solution of pentosans than that used in our experiments and hence with properties correspondingly more pronounced.

The formation of pentosan gels in dough may be affected and altered by (1) the amount and nature of the soluble pentosans, (2) the kind, amount, and manner of application of the oxidizing agent used in the dough, and (3) the amount and activity of substances in the dough that alter the viscosity or interfere with the production of gel.

A pentosan gel once formed is irreversible. Such a gel if beaten to a liquid will not reform on standing or by further oxidation. The rigidity that a gel would give to a dough would thus be broken by manipulation or handling in a bakery. Possibly an effect of remixing old dough is to break up the pentosan structure and give the dough the fluid character of younger doughs. This irreversible property of pentosans suggests that the gelling reaction in dough might desirably be delayed until all manipulations of the dough have been completed and the dough is in the pan. The rigidity that the gel formation would give the panned dough would prevent coalescence of bubbles in the proof period, and hence loss of bubble structure in the bread. Since the viscosity of pentosans is lowered and their gelling hindered or prevented by solubles from bran, germ, malted grains, or malt, it is suggested that the usual softening of dough produced by their presence or addition may be due, in part, to their effects upon the pentosans. Conversely, the most pronounced gel reaction should be obtained from well milled flours from sound wheat of less than full extraction.

There has been presented evidence that the insoluble pentosans are deposited or adsorbed upon the surfaces of the starch granules. If this is the case, it may explain why raw starch is resistant to the action of amylases. The insoluble pentosan layer may need to be penetrated before the starch itself can be attacked. This also might account for the rapid attack of amylase upon broken starch granules.

Summary and Conclusions

Flour contains approximately 1% of water-soluble pentosans that will form an irreversible gel upon reaction with certain oxidizing agents used as dough improvers.

A method of preparing the water-soluble pentosans of flour in high purity has been described.

The properties of the purified pentosans in respect to viscosity, gelling reaction, and the effect of enzymes have been shown to correspond to the same properties of flour extracts of high concentration.

Temperature change and addition of glutathione will produce effects on purified pentosan solutions that are similar to their effects upon doughs.

The gelling reaction and viscosities of soluble pentosans are much altered by extracts of malt, wheat germ, and wheat bran, but are little affected by many other dough ingredients. This may be a reason for the superior performance of well-milled refined flour from sound wheat in bread making.

The above characteristics of soluble pentosans suggest that they play an important role in controlling dough properties.

The insoluble pentosans of flour are largely associated with starch and are found in amounts proportional to the surface of the starch. This suggests a coating of insoluble pentosan on starch which may account for many of its properties and its behavior toward enzymes.

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ABILITY OF THE GLASS ELECTRODE TO DETERMINE THE TRUE pH VALUE OF BREAD DOUGHS

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In recent years the glass electrode, used in conjunction with vacuum-tube amplifiers, has become an accepted and widely used instrument for the determination of pH values. Its ability to give in moderately alkaline or acid solutions results closely agreeing with those of the hydrogen electrode has been verified by numerous observers. In making such determinations, it is only necessary to place the electrode in the solution which is then bridged directly to the calomel electrode. No stirring is necessary, no chemicals are added, and equilibrium is reached very quickly. From this simple procedure it is only a step further to insert the electrode into pastes, doughs, batters, or other semisolid substances. The development of sensitive vacuum-tube amplifiers, with their negligible current drain, allows the use of electrodes with sufficiently thick glass membranes to withstand the strain imposed upon them by such procedures.

The glass electrode was used by Landis (1934), Lanning (1936), and Shellenberger and Catlan (1938) to measure the pH values of fermenting doughs. However, none of these investigators offered appreciable data to show that the voltage of a glass electrode would be the same for a dough and a solution of like pH value. Landis presented data to show that the glass electrode when used in fermenting doughs gave values which agreed roughly with those of the ball quinhydrone electrode of Whittier and Grewe (1929). These investigators mixed quinhydrone with small amounts of the material being tested and then added sufficient water so that it could be formed into a ball. This ball was pressed around a gold electrode, which was then coupled to a calomel cell. An extract of the material tested was also made and its pH value determined with the Bailey hydrogen electrode and the capillary quinhydrone electrode. The results obtained with the ball method and the extraction method were not strictly comparable because of the dilution factor.

In order to obtain more definite information regarding the behavior of the glass electrode in doughs and solutions of like pH value, the

writer devised a method for comparing them. It is based on the assumption that if solid particles such as those of flour are suspended in a solution of a given pH value, the particles and the solution will equalize to the same pH value. If this is true or sufficiently true for practical purposes, then one could make a suspension of flour and water and after allowing time for equilibrium to be reached, the flour could be centrifuged from the water so as to form a dough in the bottom of the centrifuge cup. The pH value of the dough thus formed could then be directly compared with that of the supernatant liquid using the same electrode for both purposes. In this manner a direct comparison of the dough and solution would be made, and any appreciable variation in values might be attributed to differences of electrode action in the two mediums.

Experimental

The glass electrode used consisted of a thin membrane of Corning No. 015 glass sealed on the end of a piece of soft laboratory glass tubing approximately 10 mm in diameter and 110 mm long. The membrane was made and attached in the manner described below. A very thin-walled bulb was blown on the end of a piece of 5 mm No. 015 glass tubing. This bulb was of such thickness that the glass would spring slightly when touched with the finger. One end of the piece of soft laboratory glass tubing was heated in a gas flame until the hole was reduced to a diameter of about 5 mm. The end while still hot was pressed slowly into the glass bulb. The glass bulb readily sealed on the end of the hot tube and the excess glass was then broken off. If the tube was deformed when pressed through the glass bulb, this deformation usually indicated that the glass wall of the bulb was too thick. The outside of the tube was then coated with paraffin wax to within about one-half inch of the end on which the membrane was attached.

A calomel half cell was used for the internal electrode. Its construction was as follows: The body of the electrode was made from a piece of soft glass tubing 4 mm in diameter and about 100 mm long. One end was sealed off and the other end flared to a diameter of about 9 mm. A hole was blown in the side of the tube at a distance of about 20 mm from the closed end. A piece of platinum wire was then run through a small piece of capillary tubing about 40 mm long. A capillary melting point tube is satisfactory for this purpose, or the tube may be made by simply drawing out a piece of ordinary laboratory glass tubing. Each end of the tube was sealed and the wire cut so that about 2 mm of it projected at each seal. This tube was dropped into the prepared 4-mm tube. The 4-mm tube was then

sealed by heating in a gas flame at a point just above its side opening. The platinum wire projecting above the seal was covered with mercury into which a copper wire dipped for external connection. Pure mercury was inserted into the lower end of the tube through the small hole in its side until the lower end of the platinum wire was covered. A little 0.1*N* HCl was introduced through the side opening and then sufficient calomel to form a layer about 4 mm in depth over the mercury. The tube was then filled through the side opening with 0.1*N* HCl.

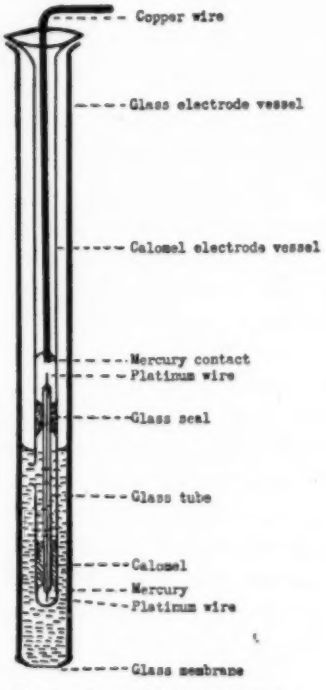

Fig. 1. Assembled glass electrode.

The tube carrying the glass membrane was filled with 0.1*N* HCl to such a depth that when the inner cell was inserted in it the side opening would be immersed in the HCl solution. In order to have the voltage remain constant, it was found necessary to move the inner electrode up and down several times a day so as to stir the HCl solution. After this stirring, the potential would sometimes vary from normal but in a few minutes would return to its normal value. The electrode was mounted on a support similar to that described by Lanning (1936). It was frequently calibrated with buffers of known pH values. A diagram of the assembled glass electrode is shown in Figure 1.

The glass electrode potentials were measured on a type K potentiometer, with a vacuum tube amplifier used as null point indicator. The circuit of this vacuum tube amplifier was similar to that described by Ellis and Kiehl (1933) and Landis (1934). The circuit was modified so as to use three Type 32 tubes instead of four Type 33 tubes as shown in the original writings. The use of these tubes greatly increased the stability of the circuit. The apparatus when used with the glass electrode herein described would indicate a potential difference of 0.2 millivolt when a galvanometer of 22 microamperes per scale division in sensitivity was used in the output stage of the amplifier.

In order to obtain dough and liquid from the same suspension for a comparison of their pH values, a series of suspensions was prepared from each kind of flour that was tested. One of these was prepared for each pH level that was used by mixing together, by means of a small electric stirrer, 30 g of flour, 100 ml of water, and the necessary alkali or acid to adjust the suspension to the desired pH value. After thorough mixing, each suspension was transferred to a 4-ounce glass-stoppered bottle and shaken occasionally. After standing at least 45 minutes, a 50-ml portion of each suspension was poured into a centrifuge tube and whirled at about 1800 rpm for 10 minutes. The flour accumulated in the bottom of the tube as a fairly firm dough. The supernatant liquid was poured off and its pH value determined by means of the glass electrode. With a spatula the dough was next removed from the bottom of the centrifuge tube and placed on a small watch glass. The excess surface liquid and the softer portion of the dough were scraped away from the more solid portion. With a sharp razor blade a cut was made directly through the firm part of the dough, with care taken to keep any remaining surface liquid from contacting the freshly cut surface. The dough was then pulled apart at the cut, and the membrane of the glass electrode, after being wet with a drop of water, was placed directly on the freshly cut surface.

Wetting of the electrode greatly inhibited the sticking of dough to it and reduced to a minimum the possibility of breakage from that cause. The dough was next bridged to the reference electrode by means of a saturated potassium chloride agar bridge, one end of which was inserted directly into the dough and the other end into a small connecting vessel filled with saturated potassium chloride solution, into which dipped the side arm of the external reference electrode. Readings were taken after one minute intervals except in the more alkaline range, where longer periods were necessary to obtain reasonably stable voltages.

Values obtained with patent flours milled from hard winter wheat and hard spring wheat are shown in Table I. These flours had an

ash content of 0.40% and a protein content of 11.9% and 11.5%, respectively. Several other flours were tested at the pH level that was naturally obtained without the addition of acid or alkali. These findings did not reveal anything different from those reported in Table I, so the results of the tests were not tabulated.

TABLE I

pH VALUES OF DOUGHS AND EXTRACTS OBTAINED FROM LIKE SUSPENSIONS

Ml of 0.1N reagent	Flour milled from hard spring wheat			Flour milled from hard winter wheat		
	pH value of solution	pH value of dough	Difference	pH value of solution	pH value of dough	Difference
6 HCl	4.68	4.78	0.10	4.67	4.79	0.12
4 HCl	5.03	5.14	0.11	4.98	5.09	0.11
2 HCl	5.48	5.60	0.12	5.40	5.52	0.12
0	5.87	5.95	0.08	5.79	5.87	0.08
2 NaOH	6.25	6.29	0.04	6.18	6.23	0.05
4 NaOH	6.63	6.59	−0.04	6.71	6.69	−0.02
6 NaOH	7.26	7.23	−0.03	7.23	7.18	−0.05
8 NaOH	7.95	7.88	−0.07	8.00	7.95	−0.05

Discussion

The results in Table I show that the maximum difference in pH values between solutions and doughs from the same suspensions did not exceed 0.12 pH unit over the range of pH values covered. At first sight this appears to be a rather large error. However, it should be remembered that the older methods involved the extraction of a dough with water, which entailed a loss of carbon dioxide and a dilution of the acid components of the dough. In the ball quinhydrone method, considerable manipulation of the dough was also necessary to knead the quinhydrone into it. It is probable that the manipulations in these methods introduced more error into the final results than the 0.12 pH difference which may exist, according to these data, in the glass electrode method.

A further study of the data indicates that the differences shown may not be due entirely to inability of the electrode to determine correctly the pH value of the dough. It should be noted that in the vicinity of pH 6.4 the pH values of the solution and dough are in good agreement. This value may represent the isoelectric point of the gluten, or in other words it may be the point where the gluten acts neither as an acid nor as a base. The tendency of the gluten salt formed by the addition of an acid or a base would be to hydrolyze back to the original value—the pH value of the isoelectric point—unless it was prevented from so doing by the presence of an excess of acid or base. Since the pH value of the original solution would then be governed by the equilibrium established between the acid or alkali

of the solution and the gluten of the suspended flour particles, it is probable that if the ratio of gluten to solution were changed, some change in pH might be expected. Such a change in concentration was, of course, effected in the dough formed by centrifugence. If such a change in pH occurred, one would expect the difference between the pH value of the centrifuged extract and that of the residual dough to be at a minimum at the isoelectric point of the gluten and to become greater as the pH value of the suspension was varied from that point. The data in Table I indicate that such a condition did exist.

From the data it may be concluded that the pH value of a dough when determined directly with the glass electrode is at least within 0.12 pH unit of the true value between pH values of 4.68 and 7.95. The differences noted appeared to be quite constant at the various pH values at which they were determined and a correction table could readily be made. However, since as before stated there is reason to believe that the differences may actually be true differences of pH value and not an electrode error, the writer does not believe that it would be desirable to make such corrections and so would recommend accepting the values given by the glass electrode when determining the pH value of dough.

Summary

The construction of a glass electrode suitable for use in doughs has been described.

Its accuracy was verified by determining the pH value of doughs and solutions centrifuged from flour suspensions adjusted to various pH levels.

The maximum difference encountered was 0.12 pH unit, which difference was obtained in the more acid solutions.

It was noted that the apparent error became greater as the pH value of the solution was farther removed from what appeared to be the isoelectric point of the gluten.

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EFFECT OF MOISTURE ON THE PHYSICAL AND OTHER PROPERTIES OF WHEAT. III. DEGREE, DURATION, AND NUMBER OF WETTING TREATMENTS[1]

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The work reported in this paper is a continuation of that previously reported (Swanson, 1941), and it involves similar procedures of wetting, drying, and subsequent testing. The main objectives of the current studies were to determine the effects of the following factors: (1) duration in days of the wetting period, together with the amounts of wetting; (2) increasing and decreasing the amount of wetting, the grain being dried between wettings; and (3) comparison of the effects of wetting Tenmarq and Chiefkan.

For the work under (1) and (2), Tenmarq wheat harvested from the general field on the Agronomy farm at the Kansas Agricultural Experiment Station was used. This was threshed from shocks and the wheat had not been exposed to any wetting by rains after it was ripe. For the Tenmarq and Chiefkan comparisons (3) there were made available by the Kansas Wheat Improvement Association one sample of each variety from the crop of 1940, and also one sample of each variety from the crop of 1941. None of these samples had been exposed to rain after maturity as far as could be judged by appearance.

Effect of Amounts and Duration of Wetting—Objective 1

Wheat samples for wetting and drying treatments weighed 1800 grams each, providing 1500 grams for milling, and one sample each for grain judging, for internal texture counts, and for reserve. For this work 38 samples were prepared and placed in gallon bottles. These were then divided into seven groups of five each, with three checks in which the moisture was 11%. No water was added to the checks but was added to five bottles of each group so as to have moisture percentages of 14, 17, 20, 23, and 26 for each group. The previous test (Swanson, 1941) had indicated that wetting up to 26% in steps of 3% was sufficient for the purposes. The weighed wheat portions were placed in the bottles and wetted at the same time. Six groups of bottles, each having the five moisture levels with the two checks, were subjected to the laboratory temperature during July; and the seventh group, with bottles at each moisture level with one check, was kept for six days in a cold storage room at a temperature of about 45°F. Of those wetted

[1] Contribution No. 91, Department of Milling Industry.

and retained in the laboratory one group of five bottles, having one bottle at each one of the moisture levels, was emptied after one day, another group after two days, and so on up to six days. The seventh group, which had been in cold storage, was also emptied at the end of the sixth day.

TABLE I

TEST WEIGHTS OF WHEAT AS AFFECTED BY AMOUNT AND DURATION OF WETTING AND TEMPERATURE DURING WETTING

Moisture to which wetted	Before drying				Dried			
	Days wetted			Average	Days wetted			Average
	1	6	6 at 45°F		1	6	6 at 45°F	
%	lbs	lbs	lbs	lbs	lbs	lbs	lbs	lbs
11 check	59.6	59.5	59.2	59.51	59.5	59.3	59.5	59.44
14	55.7	56.2	56.5	56.20	56.6	57.1	57.2	56.97
17	53.1	53.6	54.0	53.41	55.5	56.0	56.0	55.71
20	50.8	51.4	51.0	50.80	55.1	55.1	54.8	54.69
23	49.2	49.7	49.7	49.53	54.3	54.4	54.7	54.09
26	48.6	49.1	49.2	48.87	53.7	53.9	53.2	53.17
Decrease:								
Total	11.0	10.4	10.0	10.64	5.8	5.8	6.3	6.27
Av for each percentage	0.73	0.69	0.67	0.71	0.39	0.39	0.42	0.42

Moisture to which wetted	Cleaned				Scoured			
	Days wetted			Average	Days wetted			Average
	1	6	6 at 45°F		1	6	6 at 45°F	
%	lbs	lbs	lbs	lbs	lbs	lbs	lbs	lbs
11 check	59.6	59.4	59.4	59.51	62.2	61.9	62.0	62.03
14	57.3	57.6	57.6	57.56	60.9	60.9	60.9	61.00
17	56.2	56.5	56.7	56.53	60.1	59.8	60.4	60.00
20	55.8	55.8	55.7	55.57	59.5	59.4	59.5	59.30
23	55.3	54.9	55.7	55.04	59.0	56.7	59.5	58.60
26	54.8	54.7	54.8	54.47	56.7	58.7	58.7	58.20
Decrease:								
Total	4.8	4.7	4.6	5.04	5.5	3.2	3.3	3.83
Av for each percentage	0.32	0.31	0.31	0.34	0.37	0.21	0.22	0.26

Effect on test weight: The test weight of the wet wheat was obtained as soon as it was poured from a bottle. The wheat was then placed in shallow paper boxes and exposed until air-dry. Since the amount of wheat put into the bottle was known, it was a simple matter to determine when the wheat was dried near to the original 11% moisture. The test weight was then obtained on the redried wheat and samples were taken for milling, grain judging, and internal texture

counts. Immediately before milling, the test weights were retaken after the samples had passed through the laboratory cleaner. The test weights were also taken after scouring. An examination of the complete data showed that there was no significant trend in the test-weight data obtained from 1, 2, 3, 4, 5, and 6 days of wetting in the laboratory. In Table I are shown the test weights obtained after 1 and 6 days of wetting in the laboratory and the 6 days of wetting at 45°F. The figures in the "average" columns are based on the data for all groups kept in the laboratory and include the samples stored 6 days at 45°F.

The duration of the time of wetting apparently affected the test weight only slightly, probably because the maximum swelling takes place during the first day and after that there is little change. The effects of storage at the 45°F temperature differed but little from the laboratory temperature effects.

Decrease in test weight caused by wetting: Decreases in test weights for each additional 3% of wetting were greater for the smaller amounts of wetting than for the larger as shown in Table II. Those data were

TABLE II

PROGRESSIVE DECREASES IN TEST WEIGHT PER 3% INCREMENTS IN WATER USED IN WETTING

Moisture to which wetted	Decreases in test weights			
	Wet	Dry	Cleaned	Scoured
	lbs	*lbs*	*lbs*	*lbs*
14	3.31	2.47	1.95	1.03
17	2.79	1.26	1.03	1.00
20	2.61	1.02	0.96	0.70
23	1.27	0.60	0.53	0.70
26	0.66	0.92	0.57	0.40
Total	10.64	6.27	5.04	3.83

obtained by subtracting each average figure in the last column of Table I from the preceding average. The greatest decrease occurred between 11% and 14% moisture and for each additional 3% increment, the proportionate decreases in test weight were smaller. The decreases result from two factors: swelling of the kernel as a whole and loosening and roughening of the bran coat. The latter factor impedes the sliding of the kernels on each other, resulting in less dense packing. The outer layers of bran are distended by wetting, causing the kernels to occupy more space. The removal of the loosened bran coat by scouring increases the test weights. The differential decreases for each 3% increase in wetting were less for the dry than for the wet, and progressively less for the cleaned and scoured.

Increase in test weight from drying, cleaning, and scouring: The increases in test weights of the samples from different treatments are shown in Table III. These data were obtained by computing the differences in test weights after drying, cleaning, and scouring. The increases in test weights after drying were due to loss of water which has approximately two-thirds the specific gravity of the dry substance in wheat (specific gravity about 1.48). The larger specific gravity in the dry matter of wheat as compared with water explains not only the differences between the test weights of the wheat before and after drying, but also why the differences are larger with the higher percentages

TABLE III

INCREASE IN TEST WEIGHT OBTAINED BY DRYING, BY CLEANING AND BY SCOURING

Moisture to which wheat was wetted	Increases from		
	Wet to dry	Dry to cleaned	Cleaned to scoured
%	*lbs*	*lbs*	*lbs*
11	−0.07	0.07	2.52
14	0.77	0.59	3.44
17	2.30	0.82	3.47
20	3.89	0.88	3.73
23	4.56	0.95	3.56
26	4.30	1.30	3.73

of moisture. Most of the increase in test weight after cleaning was due to removal of some outside bran which had been loosened by the wetting, and since there was more loosening by greater percentages of wetting, there was an increased trend. The increases in test weights obtained after scouring were almost uniform, starting with the 14% wetting. The variability due to amounts of wetting was in the loosest bran removed in cleaning; the amount removed in scouring was the same for little or much wetting.

Flour yield, percentage of ash, and wheat grade: The flour yields were computed in percentages of the weights of the cleaned samples. The complete data on flour yields and ash indicated no definite trend for the different lengths of wetting or temperature variations; hence only the data for 1 and 6 days in the laboratory and 6 days at 45°F, as well as the averages of all the data, are given in Table IV. The wheat grades were determined by Martin Schuler of the Kansas City office of the Federal Grain Inspection Department, using the test weights as submitted and small samples. Neither the duration of the wetting period, the temperature, nor the amount of moisture added influenced the flour yields. The average test weight of the wheat wetted to 26% and redried decreased from 59.44 to 53.17, but this decrease of 6.27 pounds in test weight did not proportionately in-

fluence the flour yield (Table II). The ash percentages show an increasing trend, which was a little larger for the samples kept in the laboratory than for those kept at 45°F. The bran apparently became slightly more brittle as a result of the prolonged wetting.

The complete data on wheat grades showed that they were affected only by the amount of wetting and not by the duration or by the storage temperature, and hence only the average grades are given in Table IV. The amount of wetting was the important factor because of its influence on test weight.

TABLE IV

FLOUR YIELD, ASH PERCENTAGE, AND GRADE AS AFFECTED BY AMOUNT, DURATION AND TEMPERATURE OF WETTING (AIR-DRY)

Moisture to which wetted	Days wetted						Averages		Grades
	1		6		6 at 45°F				
	Yield	Ash	Yield	Ash	Yield	Ash	Yield	Ash	
%	%	%	%	%	%	%	%	%	
11	71.5	.467	69.8	.509	69.5	.509	70.73	.485	2 DHW
14	71.8	.483	71.8	.486	70.5	.551	71.00	.498	3 DHW
17	72.4	.482	70.9	.492	71.3	.477	70.86	.487	3 DHW
20	70.6	.489	72.5	.501	69.9	.479	70.99	.506	3 DHW
23	73.3	.493	72.2	.577	70.9	.509	71.93	.528	4 DHW
26	71.1	.518	72.3	.580	73.2	.533	72.44	.562	4 DHW
Av	71.75	.489	71.58	.524	70.88	.509	71.33	.511	—

Effect upon internal texture of duration and temperature of wetting: A barley kernel cutter, used in the previous investigations (Swanson, 1941), was used in studying the internal texture of the grain. It is not difficult to differentiate the sections of the kernels, which were clearly vitreous or plainly mealy or chalky. Those which represent transition from vitreous to mealy are designated as semivitreous. These had various degrees of mealiness or vitreousness but a closer differentiation than the one given did not seem to be warranted. The complete data showed no pronounced trend with the duration of wetting or of temperature and hence only the figures for the one day, six days in laboratory, six days at 45°F, and the averages of all the data are given in Table V.

There was a slightly greater decrease in the vitreous as well as an increase in the mealy condition in the samples stored 6 days in the laboratory over those stored 1 day, but 6 days at 45°F had only a little less effect than 1 day in the laboratory. The amount of wetting was the principal factor affecting the texture. The 3% increase in moisture from 11% to 14% did not produce changes as large as the subsequent

3% additions. The moisture at 14% is apparently only enough to cause a small amount of internal change in the endosperm. However, as shown in Table II, the increase in moisture from 11% to 14% produced notably greater changes in test weight than did the subsequent 3% additions.

Small additions of moisture to dry wheat seem to affect mostly the bran and have small effect on the internal structure. The structure is

TABLE V

INTERNAL TEXTURE AS AFFECTED BY AMOUNT OF WETTING, STORAGE TEMPERATURE AND DURATION OF WETTING

Moisture to which wetted	Days wetted					
	1			6		
	Vitreous	Semivitreous	Mealy	Vitreous	Semivitreous	Mealy
%	%	%	%	%	%	%
11	97	1	2	98	2	0
14	95	3	2	94	2	4
17	82	13	5	74	20	6
20	43	42	15	34	49	17
23	22	56	22	15	52	33
26	9	49	42	6	39	55

Moisture to which wetted	Days wetted					
	6 at 45°F			Average		
	Vitreous	Semivitreous	Mealy	Vitreous	Semivitreous	Mealy
%	%	%	%	%	%	%
11	99	1	0	97.6	1.3	1.1
14	96	2	2	94.7	3.3	2.0
17	91	5	4	81.1	14.0	4.9
20	56	36	8	40.4	45.1	14.5
23	24	52	24	18.7	53.3	28.0
26	18	44	38	12.0	47.7	40.3

not affected unless the moisture is sufficient to cause internal displacements or an expanding of the internal endosperm structure by the absorbed water. The adhesive forces are so strong that the water molecules are spread as adsorbed layers over the surfaces of the starch granules and the protein material. This will require more space for these materials and hence cause swelling. The absorbed water has such a vapor pressure that as soon as the kernels are exposed to air of a relative humidity comparable to that at which the wheat was originally dried, evaporation will take place until all the added water has disappeared. The structural relationship of the protein molecules and

the starch granules does not return to the former compact condition, however, leaving vacuoles large enough to influence the manner of light reflection, which causes a mealy appearance somewhat similar to that of compressed flour.

Effect of duration and amount of wetting upon mixogram characteristics: Mixograms were made from the flours milled from the wheat samples wetted to the various percentages as indicated in Table I and for the various periods. These mixograms were measured for the five characteristics given by Swanson and Johnson (1943). No definite trend was produced by either the duration or the temperature. Hence only the averages for the various moisture percentages are given in Table VI. With an increase in the amount of wetting, there was an

TABLE VI

EFFECT OF WETTING TO VARIOUS MOISTURE PERCENTAGES ON THE FIVE MIXOGRAM CHARACTERISTICS

Moisture to which wetted	Time of development	Height	Development angle	Tolerance angle	Weakening angle
%	*min*	*units*	*deg*	*deg*	*deg*
11	3.4	63	46	108	26
14	3.5	64	45	106	29
17	3.5	66	45	104	31
20	4.5	64	38	111	29
23	4.5	62	36	117	27
26	4.7	62	34	120	25

increase in the time of development, a slight decrease in the height, a decrease in the angle of development and increase in the angle of tolerance, and a slight decrease in the weakening angle. These trends are in the direction which indicate damaged samples, but in these the changes had only gone far enough to age the wheat with consequent improvement.

Baking results as affected by amounts and duration of wetting and storage temperatures: The baking was limited to two check samples and to those samples which had been wetted to 14%, 20%, and 26%. The data obtained are given in Table VII. The baking formula used was:

Flour 100 grams	Shortening 3 grams
Sugar 6 grams	Potassium bromate 3 milligrams
Salt 1½ grams	
Dry milk 4 grams	Absorption and mixing time determined by protein % and mixograms

There is some indication that the 5 and 6 days' wetting period in the laboratory caused some deterioration. For the shorter periods the large amounts of wetting seem to have been beneficial rather than detrimental. Thus the largest loaf volumes were obtained from wetting

to 26% for 2, 3, and 4 days but longer periods reduced the volumes. Storing at 45°F seemed to have but little effect since the loaf volumes for the 11%, 20%, and 26% wetting were nearly the same. The textures and crumb colors do not show any definite trends. A few determinations were made of the maltose values, but they showed no consistent trend.

TABLE VII

BAKING RESULTS AS AFFECTED BY AMOUNT AND DURATION OF WETTING AND STORAGE TEMPERATURES

Days wetted	Moisture content	Flour protein	Wheat grade	Baking		
				Loaf vol	Texture	Crumb color
	%	%		cc	%	%
1	11	11.9	2 DHW	855	85–0	85 c-y
1	14	12.1	3 DHW	873	84–0	85 c-y
1	20	11.4	4 HW	853	84–0	84 c-y
1	26	12.0	5 HW	883	84–0	85 c-y
2	14	12.0	3 DHW	835	84–0	85 c-y
2	20	12.0	4 DHW	835	81–0	85 c-y
2	26	12.0	5 HW	930	83–0	85 c-y
3	14	12.1	3 DHW	807	84–0	83 c-y
3	20	12.2	4 HW	863	84–0	83 c-y
3	26	12.2	5 DHW	883	85–0	84 c-y
4	14	12.2	3 DHW	873	84–0	85 c-y
4	20	12.0	4 DHW	835	83–0	83 c-y
4	26	12.2	5 HW	912	84–0	84 c-y
5	11	12.2	2 DHW	875	84–0	84 c-y
5	14	12.2	3 DHW	905	83–0	85 c-y
5	20	12.1	4 DHW	860	78–0	80 c-y
5	26	12.1	5 DHW	858	80–0	84 c-y
6	14	12.1	3 DHW	805	82–0	82 c-y
6	20	12.0	4 DHW	883	83–0	84 c-y
6	26	12.2	5 DHW	860	87–0	87 c-y
				Samples stored at 45°F		
6	11	12.1	2 DHW	838	83–0	84 c-y
6	14	12.0	3 DHW	818	84–0	82 c-y
6	20	12.1	4 DHW	838	84–0	82 c-y
6	26	12.2	5 DHW	833	83–0	82 c-y

Effects of Varying the Amounts and Number of Times of Wetting—Objective 2

Effects of progressively increasing the moisture percentage and the number of times wetted, and progressively decreasing the amounts and the numbers of wettings, but drying after each wetting were determined. The general plan can be seen from the following:

Increasing the percentage of moisture and number of times wetted

Wetted to %									
11 Check		Wetted							
14	Dry	to %		Wetted					
14	Dry	17	Dry	to %		Wetted			
14	Dry	17	Dry	20	Dry	to %		Wetted	
14	Dry	17	Dry	20	Dry	23	Dry	to %	
14	Dry	17	Dry	20	Dry	23	Dry	26	Dry

Decreasing the percentage of moisture but increasing the number of times wetted

26	Dry								
26	Dry	23	Dry						
26	Dry	23	Dry	20	Dry				
26	Dry	23	Dry	20	Dry	17	Dry		
26	Dry	23	Dry	20	Dry	17	Dry	14	Dry

At the start the five samples in the first group were wetted to 14% and five in the second group to 26%. After drying, one in each group received no further treatment but the other four were rewetted in the first group to 17% and the other four in the second to 23%. This was continued according to the above scheme. The test weights, flour yields, ash percentages, grading, and internal texture percentages are given in Table VIII.

TABLE VIII

EFFECT OF VARYING THE AMOUNTS AND TIMES OF WETTING

Moisture to which wetted	Test weights dry	Flour yield	Ash	Commercial grading		Internal texture		
				Grade	Vitreous	Vitreous	Semi-vitreous	Mealy
%	*lbs*	%	%		%	%	%	%
11 (check)	59.8	69.3	.479	2 DHW	90.0	95	2	3
14	56.8	70.7	.478	3 DHW	89.0	95	3	2
14, 17	55.5	70.8	.487	4 DHW	85.0	91	5	4
14, 17, 20	54.8	71.7	.488	4 DHW	83.0	55	34	11
14, 17, 20, 23	54.1	72.0	.495	4 DHW	78.0	29	47	24
14, 17, 20, 23, 26	53.1	73.1	.559	5 HW	62.0	12	53	35
26	53.9	71.0	.508	5 HW	67.0	20	46	34
26, 23	53.6	70.8	.506	5 HW	67.0	20	44	36
26, 23, 20	53.6	69.6	.528	5 HW	50.0	16	48	36
26, 23, 20, 17	53.6	70.6	.519	5 HW	70.0	14	50	36
26, 23, 20, 17, 14	54.4[1]	70.1	.521	4 HW	72.0	23	50	27

[1] This sample was unintentionally redried more than the others.

The amount to which the wheat was wetted had a much greater influence on the test weight than the number of wettings. The samples that were wetted the first time to 26% had very little further reduction in test weight by wetting several times more to lower percentages, while those wetted to 14% at the start had to be wetted four times more and up to 26% before the maximum reduction in test weight was

reached. It is evident that when wheat has once been wetted so as to cause maximum or near maximum amount of swelling, subsequent smaller wettings will have but little or no additional effect. In a previous study (Swanson, 1939) it was noticed that the same increases in moisture did not produce equal amounts of lowering in test weights. The moisture history of the wheats used in that study was not known and apparently some wheats had previously been wetted much more than others.

The commercial grading correlates with the test weights, but flour yields do not, as shown in the preceding tables. The ash percentages indicate fair uniformity of milling. The internal texture percentages indicate a gradual decrease of the vitreous and increase in the mealy condition, when the amounts of wetting were gradually increased, but when the amount of wetting was at the maximum at first very little further change in internal texture occurred as a result of additional wettings.

Comparison of Wetting Effects on Tenmarq and Chiefkan—Objective 3

Wheat samples made available by the Kansas Wheat Improvement Association were used in this study. Weighed portions of 1800 grams were wetted to the same percentages as in the preceding experiment on duration of wetting and then dried. This wetting and drying was repeated 4 times. The test weights, wheat grades, and internal texture percentages of the samples obtained after the fourth wetting are given in Table IX. The decrease in test weight follows the same general trend as in the trials on the duration of wetting. The total decrease in test weight was somewhat larger for both wheats from the 1941 crop than from the 1940 crop. This may have been due to the differences in moisture history, of which no record was available. The total decrease in test weight was greater for Tenmarq than for Chiefkan in the 1940 samples. In the 1941 samples, the decreases were slightly greater for Chiefkan.

The internal-texture percentages of the wetted samples of Tenmarq and Chiefkan show a greater decrease in the vitreous and a larger increase in the mealy percentages of Tenmarq than of Chiefkan in the samples of the 1940 crop, thus correlating with the decreases in test weight. However, the check sample of Tenmarq had also a smaller percentage of vitreous kernels than that of Chiefkan. For the 1941 samples the original condition was nearly the same and the internal texture counts do not show much difference between the two varieties.

Flour yield and percentage ash: Flour yields with ash percentages are given in Table X. From the figures in Table IX and those in Table X

TABLE IX

TEST WEIGHT AND INTERNAL TEXTURE OF TENMARQ AND CHIEFKAN, EACH WETTED FOUR TIMES

Moisture to which wetted	Tenmarq 1940 crop					Tenmarq 1941 crop				
	Test wt	Grade	Vitreous	Semi-vitreous	Mealy	Test wt	Grade	Vitreous	Semi-vitreous	Mealy
%	lbs		%	%	%	lbs		%	%	%
11 (check)	59.2	2 DHW	86	3	11	59.4	2 DHW	98	2	0
14	57.0	3 DHW	73	16	11	58.0	2 DHW	95	4	1
17	55.8	4 HW	40	48	12	54.7	4 HW	76	12	12
20	55.3	4 HW	17	42	41	54.2	4 HW	33	41	26
23	54.7	4 HW	16	41	43	53.1	5 HW	13	46	41
26	53.9	5 HW	6	43	51	52.6	5 HW	11	56	33
Moisture to which wetted	Chiefkan 1940 crop					Chiefkan 1941 crop				
	Test wt	Grade	Vitreous	Semi-vitreous	Mealy	Test wt	Grade	Vitreous	Semi-vitreous	Mealy
%	lbs		%	%	%	lbs		%	%	%
11 (check)	60.3	1 DHW	95	1	4	61.8	1 DHW	97	3	0
14	58.4	2 DHW	90	6	4	56.0	3 DHW	92	4	4
17	57.2	3 DHW	84	4	12	56.8	3 DHW	78	14	8
20	56.6	3 DHW	49	27	24	55.9	4 DHW	38	44	18
23	56.0	3 HW	41	33	26	55.1	5 HW	13	47	40
26	55.6	4 HW	25	36	39	54.5	4 HW	15	48	37

TABLE X

FLOUR YIELD AND ASH OF TENMARQ AND CHIEFKAN, EACH SAMPLE WETTED FOUR TIMES

Moisture to which wetted	Tenmarq				Chiefkan			
	1940 crop		1941 crop		1940 crop		1941 crop	
	Yield	Ash	Yield	Ash	Yield	Ash	Yield	Ash
%	%	%	%	%	%	%	%	%
11 (check)	69.1	.556	69.8	.491	69.9	.434	69.8	.497
14	69.2	.437	69.4	.457	73.0	.446	70.4	.471
17	69.3	.476	70.0	.474	72.7	.442	70.1	.465
20	68.9	.436	70.3	.485	71.4	.436	69.2	.456
23	69.9	.448	69.6	.532	72.3	.439	70.1	.469
26	70.1	.465	72.3	.580	71.7	.472	70.0	.480
Av	69.4	.470	70.07	.503	71.83	.445	69.3	.473

it is apparent that the flour yields were not decreased with the lowering in test weights. For the 1940 crop the test weights of the Chiefkan samples were higher than Tenmarq and the flour yields were also higher. For the 1941 crop the test weights of the Chiefkan samples

TABLE XI

AVERAGES OF MIXOGRAMS OBTAINED ON SAMPLES OF TENMARQ AND CHIEFKAN WETTED TO THE AMOUNTS GIVEN

Moisture to which wetted	Time of development	Height	Development angle	Tolerance angle	Weakening angle
%	*min*	*units*	*deg*	*deg*	*deg*
		TENMARQ			
11	3.3	64	40	118	23
14	3.3	65	37	122	21
17	3.4	63	37	122	21
20	3.5	65	36	123	22
23	4.0	60	31	130	19
26	4.0	61	34	125	22
		CHIEFKAN			
11	1.9	74	64	79	38
14	2.0	73	62	79	39
17	1.9	73	64	77	39
20	2.0	74	64	78	40
23	2.1	73	60	79	41
26	2.2	72	58	84	38

were also higher than Tenmarq, but the flour yields were nearly the same for both wheats.

Mixograms of Tenmarq and Chiefkan wetted various amounts: Mixograms were made on the flours obtained from Tenmarq and Chiefkan samples wetted various amounts and the only definite trend observed was on the basis of amounts of wetting. Hence the measurements of the various curves obtained from each variety were averaged for each moisture percentage to which it was wetted. The averages are given in Table XI.

The marked differences between Tenmarq and Chiefkan are apparent. The effects of wetting are similar for both wheats, however;

TABLE XII

BAKING RESULTS OF TENMARQ, 1941 CROP, EACH SAMPLE WETTED FOUR TIMES

Percentages to which wetted	Protein	Baking		
		Loaf volume	Texture	Crumb color
	%	*cc*		
11	12.1	833	84–0	83 c-y
14	12.3	853	87–0	85 c-y
17	12.0	858	83–0	80 c-y
20	12.0	840	83–0	82 c-y
23	12.1	888	83–0	83 c-y
26	12.1	917	83–0	84 c-y

both increase in time of development, decrease in the angle of development, increase in the angle of tolerance, and decrease slightly in weakening angle. The changes are somewhat greater for Tenmarq than for Chiefkan.

Baking tests: The baking results, together with flour protein of 1941 Tenmarq samples, are given in Table XII. Each wheat sample was wetted four times to the percentages given. The data in Table XII show that the best loaves were obtained from the samples wetted the most, and this agrees with results presented in the preceding tables.

Summary and Conclusions

Results of the following studies have been presented: (1) wetting wheat to various percentages and prolonging the wetting period from 1 to 6 days at laboratory temperatures and for 6 days at 45°F; (2) increasing and decreasing the percentages at which wheat was wetted, drying between wettings; and (3) comparison of the effects of wetting Tenmarq and Chiefkan wheats.

The effects of these various wettings were noted in terms of test weights, official grain grading, milling for flour yield, changes in internal texture, making mixograms (curves on the recording dough mixer), and baking tests on selected samples.

The test weights decreased progressively with the increased amounts of wetting, but the rate of decrease was larger with the first additions of water than with the later. Neither the duration of storage nor the 45°F storage temperature had any marked influence. The amount to which wheat was wetted had more influence on the decrease in test weight than the number of times wetted. The comparisons between Tenmarq and Chiefkan were inconclusive.

The decrease in test weight from wetting is caused by loosening the bran coat and the swelling of the endosperm. Both of these cause the kernels to occupy more space. The swelling of the endosperm changes the internal texture from vitreous to semivitreous and mealy and this change becomes greater with the increased amounts of moisture but is affected very little by the duration or the 45°F temperature of wetting.

The flour yield was not lowered by the amounts of wetting, nor by the duration or the 45°F storage temperature after wetting. The best baking results were obtained from the samples wetted the most for short periods. The 45°F temperature had but little effect on baking values at any of the levels of wetting.

Wetting affected mixing properties of the flour. The average of the measurements of the mixograms showed an increase in time of development, a slight decrease in height, a decrease in the angle of development, and weakening and an increase in the angle of tolerance.

These trends are in the same direction as those observed in studies of damaged wheats, but in most of these samples the changes had not proceeded far enough to injure the baking results.

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DEFATTING PROCEDURE FOR CORN STARCH

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In a previous communication (Kerr and Trubell, 1941) a method was described for preparing a component of corn starch, provisionally called "gamma-amylose." While it was pointed out that the use of the procedures given for certain stated reasons did not result in a quantitative yield of the amylose, one variable at least was not discussed inasmuch as its significance was not fully appreciated at the time. It might be anticipated that by following the procedures given a yield of about 5% of gamma-amylose should result. It is now known (T. J. Schoch, E. J. Wilson, Jr., and C. S. Hudson, private communication) that a variation in the residual fatty material in corn starch may cause a variation in yield of insolubles after enzymolysis, from possibly 3% to as high as 10%.

We have found that the yield of insoluble gamma-amylose obtained by treating corn starch with beta-amylase varies with the fat content of the original starch. The results of our test are given.

TABLE I

VARIATION IN YIELD OF GAMMA-AMYLOSE WITH THE FAT CONTENT OF THE STARCH

Starch	Fat	Yield of gamma-amylose
	%	% *of original starch*
Native corn starch	0.920	10.70
Corn starch, methanol extracted	0.252	8.43
Corn starch, methanol extracted	0.190	3.75

These comparative treatments were performed in a manner previously given (Kerr and Trubell, 1941) using a 10% suspension of starch in water and preheating the suspension to 95°C for 90 minutes

at pH 6.2 to gelatinize the starch. The percent of fat in the original starch was determined by the A.O.A.C. method discussed below.

Since the recovery of unexpectedly low yields by other workers might cast some doubt on the principal conclusion drawn, namely that corn starch is heterogeneous in the sense that it contains at least two component amyloses, it was deemed advisable to point out this influence of residual fatty material.

Accordingly, our preferred procedures are now given for defatting corn starch so that in subsequent treatment yields of gamma-amylose will be of the order of magnitude obtained by us.

Experimental

One kilo of corn starch at 10% moisture is shaken with 2500 ml of 85% (by weight) methanol-water solution in a flask fitted with ground glass joints to a reflux condenser. The mixture is then refluxed, by boiling, in a steam cone for one hour. It is then filtered on a Büchner funnel with suction while still hot. The cake is broken up in fresh 85% methanol, making up the volume to that of the original suspension. In this manner the extraction by refluxing for one-hour periods is repeated three additional times. After the last filtration the cake is washed by suspension in three liters of water; if necessary the pH of the suspension is adjusted (with NH_4OH or HCl) to about pH 6.0. It is finally filtered and oven-dried at about 50°C.

By such a procedure, corn starch may be reduced in fat content from approximately 1% to a value between 0.15% and 0.20% fat, as determined by the customary and convenient A.O.A.C. procedures of starch hydrolysis and fat recovery by solvent extraction of the hydrolysate. If fat determinations are made by the less frequently used method of Sherman (1932), the defatted starch as prepared above will analyze approximately 0.03% fat.

The effect of traces of fatty material (a controlling factor in the isolation of gamma-amylose from a conversion mixture) in altering the solubility and colloidal stability of components such as gamma-amylose is not only significant in isolation procedures designed to determine the composition of starch, but is also important in the chemical characterization of the fractions isolated. This line of study is being pursued further in our laboratories.

Summary

Procedures are given for defatting corn starch to a fat content of approximately 0.15% to 0.20% as determined by solvent extraction of fat after acid hydrolysis of the defatted starch.

The significance of residual fat in corn starch is discussed in relation to the solubility and colloidal stability of one of its components.

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DOUGH OXIDATION AND MIXING STUDIES. V. CORRELATION BETWEEN PROTEASE ACTIVITY, REDUCING MATTER, AND OXIDIZING EFFECTS IN DOUGH

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At least three different factors may be involved in the effects of oxidation in dough: (1) direct action on the gluten, (2) inhibition of proteolytic activity by action on the enzyme or by action on the substrate so as to reduce its susceptibility to enzymic degradation, and (3) oxidation of reducing matter.

That all of these factors may be operative has been indicated in earlier papers in this series (Freilich and Frey, 1939, 1941). The first factor was shown as a distinct possibility when harmful "excess-bromate" effects were obtained after the effects of added papain had been completely inhibited. That the second and third factors were involved was shown by experiments in which the effects of added protease or added reducing matter were overcome by bromate or by mixing the doughs in oxygen. However, the relative importance of these factors in different flours and under different conditions remains to be established.

The work presented here was undertaken with the object of making quantitative measurements of the relative amounts of the native protease activity and native reducing matter in different grades of flour, in order to see whether the values so obtained were actually correlated with improvements in baking value due to oxidation. Such measurements also presented the possibility of distinguishing between the two factors as to their relative importance.

Effects of Oxidation on Loaf Volume

Two series of flours were used. One was a group of the different grades of flour milled from the same Texas wheat, and the other a similar group of flours milled from the same Northwestern wheat.

Doughs made from the different flours were mixed in nitrogen and oxygen; doughs with added bromate were also mixed in nitrogen. The loaf volumes obtained in baking tests with doughs so treated are shown in Table I.

The data in Table I show (1) that there were marked increases in volume due to oxygen and bromate, particularly with the clear and low-grade flours, and (2) that the volume increases were greater for

TABLE I

LOAF VOLUMES FOR DOUGHS MADE FROM THE DIFFERENT GRADES OF FLOUR MILLED FROM THE SAME TEXAS AND NORTHWESTERN WHEATS

(Doughs mixed in nitrogen or oxygen 3 minutes; doughs with bromate mixed in nitrogen 3 minutes; Hobart-Swanson mixer used, modified for mixing in closed chamber; Freilich and Frey, 1939.)

Flours	Mixed in nitrogen	Mixed in oxygen	Mixed with bromate (6 mg) in nitrogen
	cc	*cc*	*cc*
From the same Texas wheat			
80% patent	1970	2000	1980
100% straight	1980	2030	2030
15% clear	1970	2080	2080
5% low grade	1680	1880	1930
From the same Northwestern wheat			
Patent	2010	2070	2030
Straight	2040	2080	2100
Clear	1930	2130	2150
Low grade	1630	1850	1930

the flours from the Northwestern wheat. The improvements in color and texture were similarly much greater with the clear and low-grade flours. Having thus obtained marked improvement due to oxidation in baking tests with these flours, we undertook the measurement of protease activity and reducing matter in the flours.

Measurement of Protease Activity by Use of the Formol Titration

That the formol titration procedure might be used to measure autolytic protease activity in dough was indicated by Freilich and Frey (1939). In that work the measurements were made on bread extracts. The recent appearance on the market of the Waring Blendor, a high-speed stirrer capable of breaking up dough into uniform suspension, made it possible to use dough extracts directly. The procedure used was as follows: 120 g of dough and enough water to give a total volume of 400 ml were stirred in the Waring Blendor for two or three minutes. The resulting suspension was centrifuged, and 50-ml portions of the centrifuged liquid were used in making the formol titrations.

Titrations on extracts from a dough without yeast, made with 300 g

of flour, 50 mg of papain, and mixed in nitrogen, gave results as shown in Table II.

The figures showed increases in formol nitrogen roughly equivalent to 1 ml of 0.5 *N* NaOH per hour. The marked differences thus observed indicated that much smaller differences, due to comparatively slight protease activity, could probably be measured with a fair degree of accuracy.

TABLE II

FORMOL TITRATIONS ON EXTRACTS OF DOUGH WITH ADDED PAPAIN
(No yeast in dough; phenolphthalein used as indicator)

	Formol (ml 0.5 *N* NaOH)	Increase in formol (ml 0.5 *N* NaOH)
At start	3.65	—
After 5 hrs	7.90	4.25
After 7½ hrs	11.30	7.65
After 27 hrs	28.00	24.35

In making titrations with extracts of a low-grade flour, it was found very difficult to obtain accurate end points colorimetrically, because of too much color in the original extract. The use of a glass electrode pH meter for titrations with such extracts avoided this difficulty and gave much more accurate results; all subsequent titrations were therefore made accordingly, with pH 8.5 as the end point.

Measurement of Protease Activity in Fermenting Doughs

Formol titrations with extracts of fermenting dough had shown great decreases in the formol values with increasing fermentation time. This was evidently due to the utilization of the soluble nitrogen constituents as food material by the actively fermenting yeast.

The effects of yeast fermentation in this respect may be illustrated by the following experiments: An excess of NH_4Cl was added to two doughs, one with and the other without yeast. Papain was added to both doughs. Formol titrations were made at the start, and after

TABLE III

EFFECTS OF YEAST ON CHANGES IN FORMOL NITROGEN AND ON PROTEOLYSIS IN DOUGH
(NH_4Cl—300 mg. Papain—20 mg per dough made with 300 g flour)

Dough	Formol [1]		Condition of dough after 21½ hrs
	At start	After 21½ hrs	
Without yeast	11.65	18.45	Very soft
With 1½% yeast	11.1	3.6	Much more fluid than dough with no yeast

[1] Formol figures in this and in subsequent tabulations are expressed as ml of 0.5 *N* NaOH.

21½ hours. At the end of this period it was noted that the yeast dough was very much more fluid than the one without yeast, but the formol figures showed a marked decrease in the yeast dough, and an increase in the one without yeast. The results are shown in Table III.

Because of this great depletion of soluble nitrogen constituents from fermenting dough, it was apparent that in order to obtain formol figures which are comparable, the measurements should be made either in doughs which are not fermented, or in doughs with exactly the same amount of fermentation.

Formol measurements in doughs with and without papain, in which increasing amounts of yeast were used, indicated that significant differences in fermenting doughs may be observed under certain conditions. The doughs were mixed in nitrogen, allowed to stand at 86°F for exactly 2 hours, at 100°F for exactly 1 hour, and were then baked at 410°F for 30 minutes. Formol titrations were made on extracts of the bread crumb. The results obtained are shown in Table IV.

TABLE IV

FORMOL NITROGEN IN DOUGHS WITH AND WITHOUT PAPAIN, WITH VARYING AMOUNTS OF YEAST

	Formol		
Percent yeast	Doughs without papain	Doughs with 20 mg papain	Differences in formol due to papain
%			
0.10	3.62	4.47	0.85
0.25	3.02	4.10	1.08
0.50	2.50	3.51	1.01
1.00	2.15	3.15	1.00

These results show that there were decreases in formol with increasing amounts of yeast, but that the differences in formol due to papain remained fairly constant, regardless of the amount of yeast used.

Table V and Figure 1 show the results of tests in which increasing

TABLE V

LOAF VOLUME AND FORMOL FIGURES FOR DOUGHS WITH INCREASING AMOUNTS OF PAPAIN

(Yeast, 1½%; dough time, 2 hrs; pan proof time, 51 to 55 min)

Papain	Loaf volume	Formol
mg	*cc*	
None	2100	2.35
5	2070	2.57
8	2060	—
11	2050	—
14	2030	2.95
17	2010	—
20	1920	3.35

amounts of papain were used in doughs with the same amounts of yeast; formol titrations were made on extracts of the bread crumb. From these results it is evident (1) that the effects of relatively small

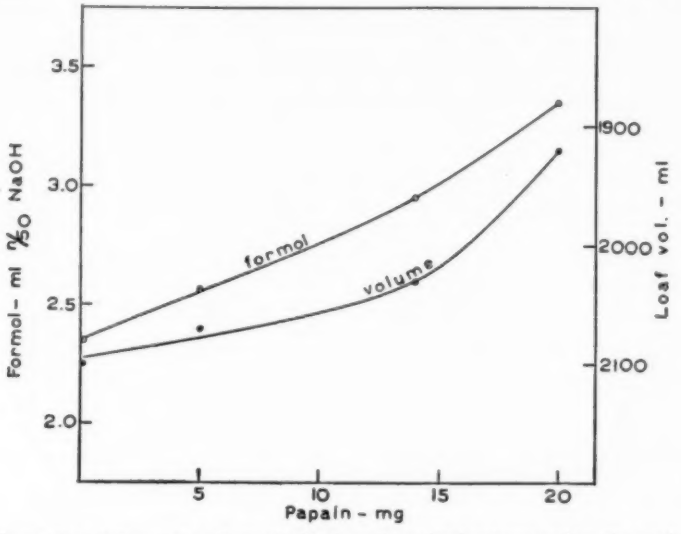

Fig. 1. Comparison between the effects of added papain on loaf volume and on the production of formol nitrogen in fermented doughs.

increments of papain may be measured by the formol titration, and (2) that an increase in formol is correlated with a corresponding decrease in loaf volume.

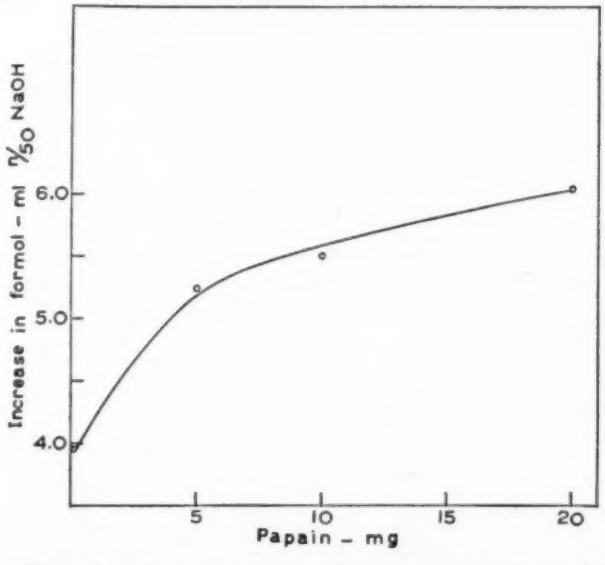

Fig. 2. Effects of papain on the production of formol nitrogen in unfermented doughs.

Measurement of Protease Activity in Unfermenting Doughs

To note the changes in formol in doughs without yeast, titrations were made on doughs with increasing amounts of papain which were allowed to stand overnight. The results are shown in Table VI and Figure 2. The increases in formol varied as the amounts of papain, indicating that the formol titration might be used to measure significant differences in unfermenting doughs.

TABLE VI

FORMOL NITROGEN IN UNFERMENTED DOUGHS WITH VARYING AMOUNTS OF PAPAIN

Papain	Formol		
	Original	After 23 hrs	Increase in formol
mg			
None	4.70	8.65	3.95
5	4.72	9.95	5.23
10	4.50	10.00	5.50
20	4.75	10.80	6.05

Effects of Octyl Alcohol on Fermentation and on Protease Activity

Because greater differences in formol were obtained when nonfermenting doughs were used (compare Tables IV and VI), and since difficulties due to varying rates of fermentation in different grades of flour might be encountered, it was decided to use doughs without yeast in formol titrations on the Texas and Northwestern wheat flours for which baking results were reported in the early part of this paper. The flours used were not sterilized.

Formol titrations were made accordingly; the results, as expected, indicated much more protease activity in the clear and low-grade flours from both wheats; but, contrary to expectations based on the baking results, the flours from the Northwestern wheat showed smaller increases in formol than the Texas flours. It had been observed that after standing overnight the Northwestern flour doughs showed a slight amount of fermentation, probably due to contamination with minute traces of yeast; it appeared probable that the slight fermentation removed sufficient amounts of formol nitrogen from the doughs to cause the unexpected results. It therefore seemed advisable to try a fermentation inhibitor in the doughs.

It had been found in the course of some other work that octyl alcohol retarded fermentation to a marked degree. Experiments were therefore conducted to observe the effects of octyl alcohol on fermentation and on protease activity. Octyl alcohol was used in doughs with 2% yeast and 50 mg of papain. The doughs were allowed to stand at 86°F for 3 hours, then baked. Formol titrations were made on ex-

tracts of the bread crumb, prepared by stirring in the Waring Blendor and centrifuging. Fermentation rate was indicated by increase in dough volume. Table VII shows the results obtained.

TABLE VII

EFFECTS OF OCTYL ALCOHOL IN DOUGHS WITH 2% YEAST AND 50 MG PAPAIN PER 300 G FLOUR

Octyl Alcohol	Fermentation	Formol
ml		
0	Normal	6.70
6	None	15.35
12	None	15.95

The results showed (1) that 6 ml of octyl alcohol was sufficient to completely stop the fermentation in a dough with as much as 2% yeast, and (2) that octyl alcohol did not inhibit protease activity. (The low formol figure for no octyl alcohol is, apparently, due to the utilization of the formol nitrogen by the fermenting yeast.)

Protease Activity in the Flours from the Same Texas and Northwestern Wheats

With 6 ml of octyl alcohol per 300 g of flour, formol titrations were made on extracts of the doughs made from the Texas and Northwestern flours mentioned above. No yeast was added. The doughs were mixed in nitrogen and allowed to stand for 24 hours at 86°F. The results are shown in Table VIII.

TABLE VIII

FORMOL TITRATIONS ON UNFERMENTED DOUGHS

(300 g flour, 15 g sugar, 5 g salt, and 6 ml octyl alcohol used in each dough)

Flours	Formol		Increase in formol
	Original	After 24 hrs	
From the same Texas wheat			
80% patent	4.51	6.28	1.77
100% straight	5.13	7.07	1.94
15% clear	5.33	7.99	2.66
5% low grade	6.59	10.02	3.43
From the same Northwestern wheat			
Patent	4.73	7.06	2.33
Straight	5.10	7.55	2.45
Clear	5.97	9.38	3.41
Low grade	8.10	13.41	5.31

The results indicated progressively greater increases in formol nitrogen from the higher to the lower grades of flour, for both the Texas and the Northwestern wheats; the original values also varied in the same order.

The formol figures also showed definitely greater protease activity in Northwestern than in the Texas flours.

That there is definite correlation between the protease activity in the flours and the improvement in volume produced by oxidation, is shown in Table IX; it is evident that the greatest improvements in volume due to oxidation were obtained with the flours showing the greatest proteolytic activity.

TABLE IX

COMPARISON BETWEEN INCREASES IN VOLUME DUE TO OXIDATION AND INCREASES IN FORMOL FOR DIFFERENT FLOURS

Flours	Increases in volume (calculated from Table I)		Increases in formol N (from Table VIII)
	Due to oxygen	Due to bromate	
	ml	*ml*	
From the same Texas wheat			
Patent	30	10	1.77
Straight	50	50	1.94
Clear	110	110	2.66
Low grade	200	250	3.43
From the same Northwestern Wheat			
Patent	60	20	2.33
Straight	40	60	2.45
Clear	200	220	3.41
Low grade	220	300	5.31

Reducing Matter in the Different Flours

The relative values for reducing matter in these flours were determined by use of an iodine titration method which has been described in another paper (Freilich, 1941). Table X shows the values so obtained. The results showed increasing amounts of reducing matter

TABLE X

REDUCING MATTER IN DIFFERENT FLOURS

(Figures show ml of iodine solution taken up by water extracts)

	Flours from the same Texas wheat	Flours from the same Northwestern wheat
	Iodine solution (ml 0.01 N)	
Patent	0.9	0.9
Straight	1.1	1.2
Clear	1.4	1.5
Low grade	1.9	3.5

with decreasing flour grade, and more reducing matter in the Northwestern than in the Texas flours. A comparison between these figures and those in Table IX shows that there is also a definite correlation between increase in volume due to oxidation and reducing matter for the different flours. These interrelationships are shown more clearly

in Figure 3, in which increases in volume are compared to reducing matter and to increases in formol.

It seems apparent from Figure 3 that the correlations between volume increase and protease activity are similar to the correlations

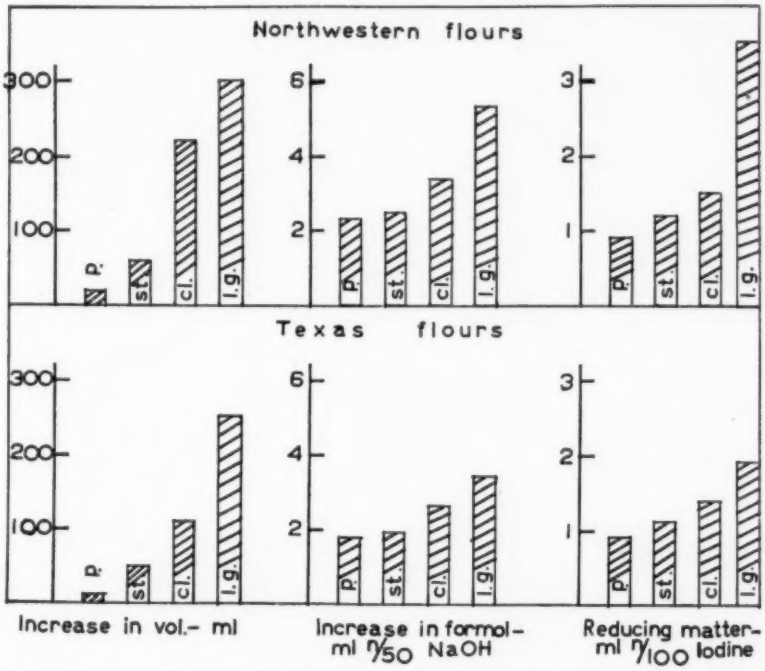

Fig. 3. Comparisons between increases in loaf volume due to oxidation (bromate), proteolytic activity—as indicated by production of formol nitrogen in dough—and reducing matter, for the different grades of flour milled from the same Texas and Northwestern wheats. Flour Grades—patent (p), straight (st.), clear (cl.), low grade (l. g.).

between volume increase and reducing matter. The indications are that both factors may be relatively important with respect to improvement in baking value due to the use of oxygen or oxidizing agents.

Discussion

On the basis of more recent work, Laitinen and Sullivan (1941) and Baker, Parker, and Mize (1942) emphasize the theory of direct action on the gluten, and are inclined to disregard or minimize other factors; the former go so far as to say that the proteolytic enzyme theory seems untenable.

There seems to be little doubt that the protease theory does not give the whole picture, yet in view of our own and other results, this tendency to discard it entirely appears unjustified. Proteolysis doubtless does have some influence, even though its contribution may not be a major one.

It is probable that no one theory is sufficient in itself; consequently work concerning the other factors involved is certainly necessary and worthwhile. The presentation of new theories emphasizes the need for evaluating the different factors on a quantitative basis.

Summary

The different grades of flour milled from the same Texas and Northwestern wheats were compared in baking tests in which the doughs were mixed in nitrogen and oxygen; doughs with added bromate, mixed in nitrogen, were also included.

The autolytic protease activity in these flours was measured by a formol titration method, with a glass electrode pH meter and titration to pH 8.5.

Significant differences in formol nitrogen due to added papain were obtained in both fermenting and unfermented doughs.

Octyl alcohol was found to inhibit fermentation without retarding protease activity.

Reducing matter in the different flours was determined by the use of an iodine titration method.

Protease activity and reducing matter were found to be of greatest magnitude in the lower grades of flour.

Definite correlations were found between: (1) improvement in volume due to oxygen or bromate and increases in formol nitrogen in the different grades of flour, (2) improvement in volume due to oxygen or bromate and reducing matter in the different grades of flour, and (3) reducing matter and protease activity in the different grades of flour. These results indicated that both reducing matter and protease activity must be considered important factors in their influence on the baking quality of flour.

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REPORT OF 1941-42 COMMITTEE ON METHODS OF TESTING CAKE FLOUR

J. W. MONTZHEIMER, *Chairman* [1]

Centennial Flouring Mills Co., Spokane, Washington

(Read at the Annual Meeting, May 1942)

The project this year was a continuation of collaborative work carried on by this committee during the past few years in an attempt to determine the type of information that may be obtained by the use of the A.A.C.C. cake formula and its supplements. This year a comparison has been made of the sugar tolerances of different cake flours, baked by the A.A.C.C. formula and a commercial type of formula with emulsified shortening. The three flours were analyzed as follows:

	Moisture	Protein	Ash	pH
	%	%	%	
E, cake flour	11.0	7.6	0.33	4.8
F, cake flour	10.7	6.84	0.34	4.9
L, fancy clear	11.	9.23	0.55	5.0

The first two flours, E and F, were commercially milled cake flours, while the third was a fancy clear taken from cake flour. All were rebolted to a fine, even texture and bleached to a satisfactory pH.

Both layer and loaf cakes were baked, by the A.A.C.C. basic formula and also with a sugar supplement of 120% as compared with flour, and a sugar supplement of 130% as compared with flour. The following commercial-type formula was chosen for making comparisons:

Flour	260 grams	Salt	10 grams
Sugar	260 "	Milk	230 "
Shortening (emulsified)	130 "	Egg whites	170 "
Baking powder	16 "		

The same three flours were baked by this formula, then with a sugar supplement of 125% as compared with flour and finally with a sugar supplement of 150% as compared with flour. We found it necessary to use a larger sugar supplement in order to test the limits of tolerance of the flours in the presence of emulsified shortening.

Figure 1 shows the loaf cakes baked with the three flours by the A.A.C.C. formula with supplements. This picture illustrates very plainly the tolerance of the flours for sugar in the A.A.C.C. formula. Figure 2 shows results with the same flours in the commercial formula and indicates that the same flours responded to the increase in sugar in

[1] This year's Cake Committee was composed of W. E. Stokes, R. Mitchell, D. Wade, L. Armstrong, W. V. Van Scoyk, A. J. King, H. V. Moss, and H. K. Murer.

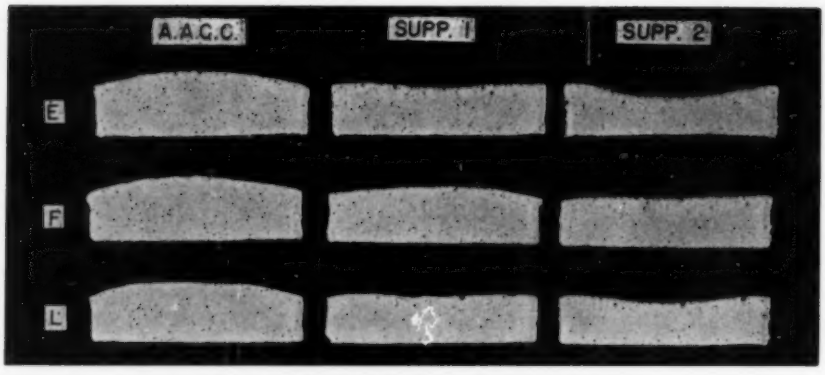

Fig. 1. Cakes baked by the A. A. C. C. formula.

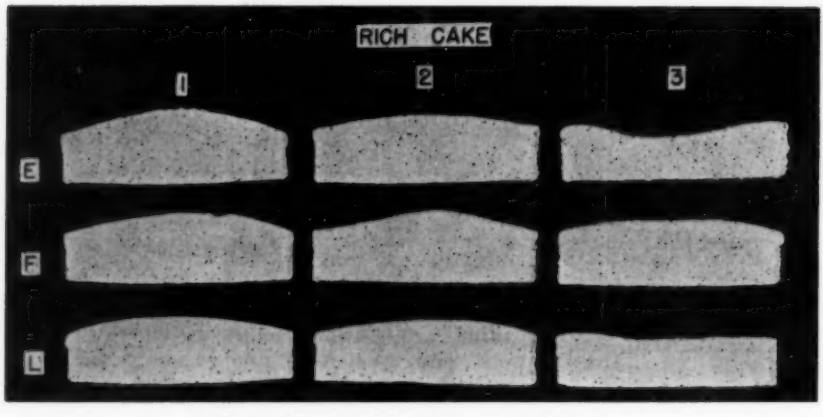

Fig. 2. Cakes baked by the commercial formula.

TABLE I

AVERAGES OF COLLABORATORS FOR FLOUR E

Formula	Sugar	Type	Specific gravity of batter	Specific volume of cake	Volume	Score	Grade
	%				cc		
A.A.C.C.	96	Layer	.904	2.89	989	82.7	2
		Loaf	.904	2.83	802	86	2
A.A.C.C.	120	Layer	.851	2.81	974	83.1	2
		Loaf	.851	2.62	750	82.2	2
A.A.C.C.	130	Layer	.891	2.51	848	69	3
		Loaf	.891	2.23	660	67.7	3
Rich type	100	Layer	.94	3.09	1013	84	1
		Loaf	.94	2.86	755	86.2	1
Rich type	125	Layer	.956	3.03	1007	87.7	2
		Loaf	.956	2.82	843	88	2
Rich type	150	Layer	.977	2.76	901	72	3
		Loaf	.977	2.47	664	70	3

much the same manner as they did in the A.A.C.C. formula. Table I gives the actual averages of the collaborators for flour E. A drop in grade from 86 to 67 as the sugar was increased represents the collaborators' scores on the decrease in cake quality as illustrated in Figure 1. Table II shows the averages of the collaborators for flour F. F showed more tolerance for sugar than E. Table III gives the averages for flour L.

TABLE II

AVERAGES OF COLLABORATORS FOR FLOUR F

Formula	Sugar	Type	Specific gravity of batter	Specific volume of cake	Volume	Score	Grade
	%				cc		
A.A.C.C.	96	Layer	.818	2.79	1032	86	2+
		Loaf	.818	2.79	800	89	2+
A.A.C.C.	120	Layer	.869	2.86	986	87.3	1
		Loaf	.869	2.71	767	89.5	1
A.A.C.C.	130	Layer	.879	2.77	947	82	1
		Loaf	.879	2.48	709	83	1
Rich type	100	Layer	.965	3.02	1012	87.4	2
		Loaf	.965	2.82	752	87	2
Rich type	125	Layer	.989	3.11	1015	87.7	1
		Loaf	.989	2.93	771	88.5	1
Rich type	150	Layer	1.00	2.91	971	83	1
		Loaf	1.00	2.73	782	82.1	1

TABLE III

AVERAGES OF COLLABORATORS FOR FLOUR L

Formula	Sugar	Type	Specific gravity of batter	Specific volume of cake	Volume	Score	Grade
	%				cc		
A.A.C.C.	96	Layer	.834	2.71	947	74.3	3
		Loaf	.834	2.45	758	76.5	3
A.A.C.C.	120	Layer	.854	2.63	918	73.8	3
		Loaf	.854	2.45	721	74.2	3
A.A.C.C.	130	Layer	.873	2.46	851	69.8	3
		Loaf	.873	2.33	664	65.7	3
Rich type	100	Layer	.935	2.79	989	76	3
		Loaf	.935	2.82	758	76.7	3
Rich type	125	Layer	.965	2.92	965	76	3
		Loaf	.965	2.66	719	77.7	3
Rich type	150	Layer	.956	2.66	892	70	3
		Loaf	.956	2.37	635	68	3

It is the opinion of the Committee that the A.A.C.C. formula may be used for prophesying the sugar tolerance of a cake flour in different types of formulas when the proper supplements for sugar are used. In our opinion, this particular series showed more marked difference with the A.A.C.C. formula than with the commercial type of formula.

REPORT OF THE 1941-42 COMMITTEE ON TESTING BISCUIT AND CRACKER FLOURS

W. H. HANSON, *Chairman*

Commercial Milling Co., Detroit, Michigan

(Read at the Annual Meeting, May 1942)

The recommendations submitted by the 1940–41 committee indicated an urgent need for further collaborative cooky test bakes with the formula and procedure proposed by Alexander (1933). A survey of the recommendations offered by collaborators indicated that the original formula should be modified to give greater sensitivity and sharper differentiation among flours. It was therefore suggested that a few minor changes be made in the formula, and that laboratory investigations of baking procedures along these lines be given further consideration. Almost without exception, each member expressed a belief that the test is suitable and informative for the evaluating of cooky-making properties of soft winter wheat flours.

The preliminary work of the 1941–42 committee was to modify the cooky test formula, and to eliminate one or more of the ingredients which have been heretofore added so as to provide a relatively easy and informative test. As a result of this preliminary study, many worth-while recommendations were made which would not alter appreciably the formula or procedure used the previous year. At a group meeting of the committee, it was suggested by Miss Pearl Brown that the whole eggs be eliminated from the cooky test formula. This suggestion was based on the fact that eggs constitute a variable not easily controlled as to uniformity. Cooky bakes were therefore made without this ingredient, and each member reported favorably on the change.

Other features recommended as a result of the preliminary study were as follows: (1) that the amount of flour used in the formula should be computed on a 13.5% moisture basis, (2) that additional distilled water approximating 75% of total egg weight be added, (3) that all members use the same type and granularity of sugar, and (4) that ammonium bicarbonate be substituted for ammonium carbonate.

It was suggested that study this year be devoted exclusively to flours milled from the white wheat varieties, as these are usually recognized as preferred cooky types. Our study is therefore representative of those geographical areas in which the low-protein, low-viscosity flours are usually milled. It was the opinion of some of the members that even within the white wheat varieties the spread potentialities of the flours would vary considerably. Instructions to the mills supplying these flours was in accordance with the usual specifications, 0.41 to

0.42% ash, 7.50 to 8.00% protein, and a maximum viscosity of 35° MacMichael as determined by the no-time method for unbleached flours of 100% extraction.

Purpose of Study

Our study was made primarily to evaluate apparent differences in flours all milled from the white wheat varieties by cooky test bakes, and to determine which flour had the greatest spread potentialities from the calculated "spread-factor" results, and also to check the possible effect of granulation or particle size on the spread behavior. A statement on the cooky test formula and procedure, inspection records, and ingredients was included with the flour samples sent to collaborators.

Flour Characteristics

The four flours selected are referred to as A, B, C, and D, the analyses of which are shown in Table I. The formula and procedure are shown in Table II.

TABLE I

FLOUR ANALYSES

Flour [1]	Ash (15% mb)	Protein (15% mb)	Viscosity		Granulation (Through 250-mesh, 60 minutes)
			No-time	1 hour digestion	
	%	%	°*MacM*	°*MacM*	%
A	.410	8.18	37	54	85
B	.425	7.97	24	36	76
C	.398	7.80	29	42	92
D	.417	7.60	27	45	72

[1] Flour A submitted from New York State, flours B and C submitted from Michigan, flour D submitted from Pacific Coast.

TABLE II

LABORATORY COOKY TEST AND PROCEDURE FORMULA

Ingredients	Grams	Flour basis
		%
Flour (unbleached) [1]	224.0	100.00
Baker's special sugar	130.0	58.04
Hydrogenated fat [2]	64.0	28.57
Salt	2.1	0.93
Sodium bicarbonate	2.5	1.12
Distilled water [3]	19.5	8.75
Ammonium bicarbonate	0.5	0.22
Skim milk suspension [4]	40.0	17.85

[1] 224 g flour, basis 13.5% moisture.
[2] Spry at 75°F.
[3] Approximately 75% of total egg weight.
[4] 28.2 g of spray-powdered skim milk dissolved in 150 cc distilled water.

Procedure

The laboratory procedure was as follows: Cream sugar, shortener, and soda three minutes, cutting down after each minute (Kitchen Aid Model G, second speed 128 rpm or equivalent). Dissolve salt and ammonium bicarbonate in the skim milk suspension, and add to this the distilled water. Add this milk suspension during 1 minute in low-speed (62 rpm on Kitchen Aid, or equivalent). Scrape down. Mix 1 minute in second speed. Add whole quantity of flour, mix for 2 minutes in low speed, cutting down after each half minute.

Place small handfuls of batter at 6 well spaced points on a cooky sheet so that the cookies when cut will be about 2 inches apart. Make sure that each handful of batter is coherent, and not composed of different scraps pressed together, as this latter practice tends to produce imperfect cookies. Lay wooden strips 7 mm in thickness along each side of the cooky sheet, and roll the batter out with a rolling pin to this height. Cut a cooky in the center of each piece with the cutter provided (60 mm diameter). Remove scrap and discard, leaving the cookies in place ready to be baked.

Bake cookies at 400°F for 10 minutes. On removal from the oven immediately lift the cookies from pan to cooling rack or absorbent paper. After 30 minutes from the oven, compute the spread factor W/T. Thickness can best be measured by piling 6 atop one another, and averaging the height. The average diameter should be obtained by making two measurements, the major and minor axes of each cooky. The spread factor W/T should be computed, W being the average diameter and T the average thickness. The greater this factor the more spread possessed by the cookies.

TABLE III

CALCULATED SPREAD FACTOR OF FLOUR SERIES

Flour	Collaborator							Rank of flour
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	
A	8.97	9.37	8.04	8.30	8.275	8.87	7.13	1
B	8.81	8.27	7.69	7.80	8.168	8.33	6.28	3
C	8.00	7.39	6.75	7.20	7.472	7.52	5.57	4
D	8.87	10.23	7.87	8.17	8.254	8.48	6.35	2

The results of the calculated spread factors submitted by the collaborators seem important in that very good agreement is shown in the order of rank of the series, from the greatest to the least spread. This differentiation between almost identical flours is progressively better in many respects than that reported by the committee of last

year. The study indicates variations in the spread potentialities of flours all milled from the white wheat varieties grown in various localities of the United States. Seemingly important is the fact that the cooky bakes did not correlate with the analyses, especially the viscosity. This is shown by flour A, which had the highest viscosity, but which gave the greatest spread. Differences noted in the spread factor between flours B and C are due entirely to differences in the grinding and bolting operations in milling.

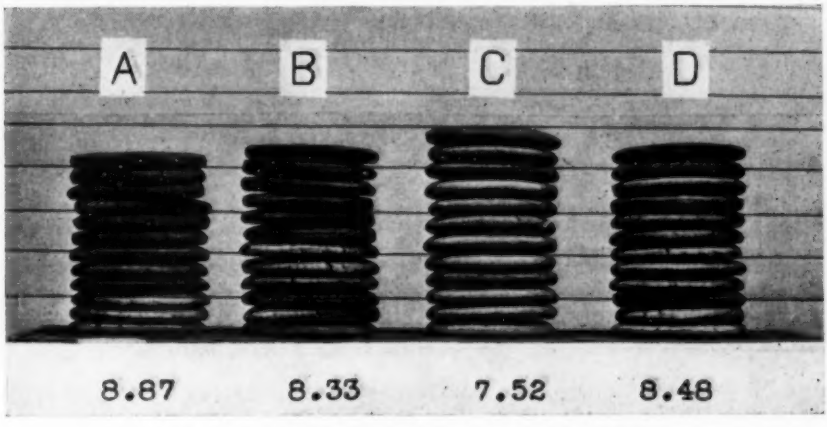

Fig. 1. Calculated spread-factor results with cookies.

Figure 1 shows the results obtained by the cooky test of the four flours selected. Comparing the relative height of the cookies, it is apparent that flour A gave the greater spread, while flour C gave the poorest. Flours B and D gave approximately the same calculated spread factor.

Recommendations

The committee recommends that further laboratory tests be made with the cooky formula and procedure, and that some consideration be given to other types of shortenings, and to variations in baking times and temperatures. It is also recommended that the committee devote another year to a study of the same flours milled from wheats of the new crop.

Acknowledgments

The chairman wishes to express his thanks to the Victor Flour Mills of Pittsford, New York, and to the Centennial Flouring Mills of Tacoma, Washington, for the flours submitted from those states. To the members of this committee, C. E. Bode, T. E. Hollingshead, F. R. Schwain, H. M. Simmons, O. P. Skaer, and Miss Pearl Brown, the chairman also wishes to express his appreciation and thanks.

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INVESTIGATION OF A DEATH BY ASPHYXIATION IN A GRAIN ELEVATOR BIN CONTAINING FLAXSEED [1]

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(Read at the Annual Meeting, May 1942)

Early in November, 1941, the death of a Twin Cities elevator employee occurred 44 hours after he had collapsed upon entering a grain storage bin containing flaxseed for the purpose of examining the condition of the grain. The bin, a concrete interstice closed tank, had been filled with 262,860 pounds of flaxseed to within about 7 feet from the top, 58 days previously, and had remained closed until the time of the incident.

The workman opened the iron cover and jumped to the surface of the grain while a partner remained on top of the tank to assist him out of the bin; in a few moments he fell forward with his face buried in the grain. His partner, after calling for help, also jumped to the surface of the grain and he likewise collapsed. Other elevator employees, equipped with ropes and safety belts, quickly removed the two men, the first within about ten minutes, and the second within about five minutes after entering the bin. Both were unconscious but the second man to enter the tank was still breathing and quickly regained consciousness. Artificial respiration soon restored the breathing of the first employee but, despite hospital treatment under an oxygen tent, he never regained consciousness and died 44 hours later.

An autopsy was conducted. The only abnormalities noted were grade 2 cyanosis in the lips, slight purplish hypostasis posteriorly, marked reduction of crepitation in the lungs, the lower lobes of which exhibited multiple nodular, palpated areas which were found to be hemorrhagic. A test of the blood for carbon monoxide was negative.

Immediately after the incident, the bin was sealed and the Division of Agricultural Biochemistry was requested to examine the air in the

[1] Paper No. 2020, Scientific Journal Series, Minnesota Agricultural Experiment Station.

bin for the presence of toxic constituents. The studies undertaken in this connection are reported in the present paper.

Investigation

The circumstances surrounding this case suggested that hydrogen cyanide might possibly have been responsible for the sudden collapse and subsequent death. The victim exhibited extreme cyanosis and the body was rigid upon removal from the bin. The flaxseed had considerable sprout damage and might have been expected to contain a relatively high percentage of the cyanogenetic glucoside-linamarin; the high moisture content at which the seed was binned would favor the enzymic release of hydrogen cyanide. This hypothesis seemed to be strengthened when a sample of the moistened flaxseed taken from the bin gave a much more intense qualitative test for hydrogen cyanide with sodium picrate paper than a control sample of sound flaxseed. The first tests were accordingly directed to the determination of the hydrogen cyanide content of the air above and within the flaxseed. Picrate-paper tests and also absorption tests, in which several liters of both overseed and interseed air were aspirated through silver nitrate and sodium hydroxide solutions, respectively, failed to reveal the presence of any free hydrogen cyanide, thus eliminating this gas as a possible cause of death.

Analyses were next undertaken to determine whether the respiratory activity of the flaxseed had been sufficiently high to produce a lethal atmosphere. Gas samples were drawn from the bin at the grain level and also from six feet below the surface and analyzed for oxygen and carbon dioxide by means of a Haldane-Henderson apparatus. The atmosphere immediately above the flaxseed was found to contain 1.8% oxygen and 11.1% carbon dioxide, while the interseed atmosphere six feet below the surface contained only 0.4% oxygen and 12.6 carbon dioxide. Since the man-hole cover of the bin had been open for some time during the rescue of the men and later to secure samples, the interseed air values are probably more nearly representative of the condition of the atmosphere above the grain at the time the accident occurred.

At this stage of our investigation, the Division of Industrial Health, Minnesota Department of Health [2] began an independent study and obtained values for oxygen and carbon dioxide in close agreement with those reported above. This organization also carried out qualitative tests for hydrogen cyanide, arsine, phosphine, and hydrogen sulfide and none of these gases was found to be present. However, 0.035% carbon monoxide was found in the overseed air by the Mines Safety Appliance Company carbon monoxide indicator, and in the overseed

[2] Private communication.

air of another tank containing flaxseed 0.020% carbon monoxide was found. Later, the Minnesota Industrial Commission,[2] employing a similar apparatus, found 0.04% carbon monoxide in the overseed atmosphere of the bin in which the death occurred.

These results show quite definitely that there was an insufficient amount of oxygen in the tank atmosphere to sustain life, and indicate that the death of the workman was caused primarily by asphyxiation resulting from the low oxygen and high carbon dioxide content. Ordinary atmospheres containing 0.02% to 0.04% carbon monoxide are considered safe for breathing for one to two hours.

It should be emphasized that death from anoxemia will occur much more rapidly when the carbon dioxide content of the oxygen-deficient atmosphere is high. The respiratory exchange of oxygen and carbon dioxide depends upon the differences in the tensions of these gases in the venous blood and alveolar air. When a normal atmosphere is breathed, the alveolar gas has a higher oxygen tension and a lower carbon dioxide tension than the venous blood, so that oxygen diffuses into the blood while carbon dioxide is lost to the alveolar air. Normally, the oxygen tension of the venous blood varies between 35 and 40 mm of mercury pressure, which corresponds to that of air containing from 4.6% to 5.3% oxygen at 760 mm mercury pressure. At oxygen contents of alveolar air below these approximate levels, oxygen will be transferred from the blood to the lungs. When the carbon dioxide concentration in the alveolar air is above normal, the carbon dioxide content of the blood is increased, thereby stimulating the respiratory center which controls the rate of respiration. As a consequence of the increased rate of breathing, the loss of oxygen from the venous blood to the lungs is accelerated and anoxemia occurs more rapidly than where breathing has been stopped as in drowning. While an increase in the amount of carbon dioxide inhaled produces an increased rate of breathing, Jacobs (1941) points out that high concentrations paralyze the respiratory center, resulting in asphyxiation and death. According to this author, 2% carbon dioxide in otherwise normal air increases the lung ventilation 50%; 3% causes a 300% increase and breathing is laborious; 10% can be endured only for a few minutes; while with 12% to 15% unconsciousness rapidly occurs and death may take place.

In the atmosphere of the flaxseed bin, the deficiency in oxygen was accompanied by a toxic percentage of carbon dioxide. Either of these could, in itself, cause death.

This case is not an isolated one. Yearsley (1921) has reported the death of an employee of a Utah milling company under very similar circumstances. The workman collapsed when he entered a covered concrete tank which had been filled with damp barley (15% moisture)

for 68 days. A second employee attempted to rescue him but also collapsed. Removal of the men from the bin was effected by the fire department, the first within about 12 minutes and the second within 8 to 9 minutes of the time they entered the bin. The former was dead but the second survived after being unconscious for 3 to 4 hours and suffering a loss of memory for 5 days. An analysis of the air showed 3.5% oxygen and 12.6% carbon dioxide.

Two additional cases were cited by Price, Roethe, and Bradshaw (1937); in one, two men were asphyxiated when they entered a steel bin filled with damp kafir corn and in the other, two men lost their lives in a tile bin (12 × 12 feet) about half filled with damp oats.

In view of the lethal atmosphere found in the fatal bin, it seemed of interest to examine the composition of the air in other closed tanks containing stored grain. The results, recorded in Table I, show that the interseed air of three of the four bins which contained flaxseed was markedly deficient in oxygen and high in carbon dioxide, while the interseed air in the four tanks containing wheat and barley was almost normal in composition.

Studies have revealed that the respiratory activity of grain varies widely and is influenced by both inherent and environmental factors. The researches of Bailey and Gurjar (1918), Bailey (1921, 1940), Coleman, Rothgeb, and Fellows (1928), and others have demonstrated that the rate of respiration increases with moisture content. At equivalent moisture contents, Bailey (1940) found that flaxseed had a much higher respiratory rate than cereal grains. This was attributed to the higher oil content of flaxseed. As the oil is immiscible with water, this results in a higher moisture content of the hydrophilic constituents than in cereal grains containing the same percentage of moisture. Cracked, shriveled, immature kernels respire more rapidly than sound, plump grain of the same moisture content; the presence of foreign material and of sprouted, frosted, or heat-damaged kernels also increases respiration. Other factors being equal, Bailey and Gurjar (1918) found that wheat respiration increased with temperature up to 55°C, whereas an accumulation of carbon dioxide in the interseed atmosphere had a depressing effect. It is now quite generally recognized that the respiration of bacteria and molds associated with grain may account for a large share of the respiratory activity exhibited in storage. Ramstad and Geddes (1942) have recently reviewed the literature in this field and have reported the results of numerous experiments with soybeans which indicate that microörganisms are the primary cause of high respiratory rates associated with heating and other damage to stored soybeans.

The lower oxygen and higher carbon dioxide content of the inter-

TABLE I

COMPOSITION OF ATMOSPHERE IN BINS CONTAINING WHEAT, BARLEY AND FLAXSEED

Bin No.	Grain	Quantity of grain in bin	Distance from top of bin to grain	U. S. Grade	Moisture [1]	Overseed air [2]		Interseed air [3]	
						Oxygen	CO_2	Oxygen	CO_2
		lbs	*ft*		%	%	%	%	%
308	Wheat	2,152,890	4	1 Hard DNS	12.3	—	—	20.3	0.1
103	Wheat	2,194,390	6	1 DNS	13.7	—	—	20.3	0.3
114	Barley	2,152,890	6	2 malting barley	12.8	—	—	20.8	0.3
309	Barley	1,753,490	2	2 malting barley	13.4	—	—	19.9	0.3
106	Flaxseed	867,650	35	1 flax	7.8	20.1	0.1	19.3	0.5
204	Flaxseed [4]	163,020	60	Sample grade flax	7.9	10.9	7.0	8.3	9.0
409	Flaxseed	390,760	6	Sample grade flax	8.1	19.6	0.1	10.1	3.3
407	Flaxseed [5]	262,860	7	Sample grade flax	9.2	1.8	11.1	0.4	12.6

[1] Determined by two-stage vacuum oven method.
[2] Samples collected at grain level.
[3] Samples collected six feet below surface of grain.
[4] Flaxseed from same lot as that stored in Bin No. 407.
[5] Flaxseed bin in which death of workman occurred.

seed air of the fatal bin, as compared with that of the other tanks containing flaxseed, suggested that conditions existed in this bin which were favorable to a high respiratory rate. Accordingly, it seemed desirable to carry out analytical studies on samples of flaxseed drawn from the four bins listed in Table I. Subsamples were also submitted to the Minneapolis office of the Grain and Seed Division, Agricultural Marketing Service, for the determination of grade and to the Minnesota State Seed Testing Laboratory for germination tests. The chemical studies included determinations of moisture, total nitrogen, combined hydrogen cyanide, oil content, iodine value, fat acidity, and respiratory activity.

Moisture content was determined by the two-stage vacuum oven method as described by Ramstad and Geddes (1942), total nitrogen and fat acidity as outlined in Cereal Laboratory Methods (4th ed., 1941), and total hydrogen cyanide formed by hydrolysis of the cyanogenetic glucosides present in the seed was determined by the alkaline titration method of the American Association of Official Agricultural Chemists (1940). Oil content was determined by extracting two-gram samples with petroleum ether (Skellysolve F) in Butt tube extractors; after two hours extraction the samples were reground with sand and the extraction continued overnight. The solvent was evaporated on a steam bath and the oil weighed after drying *in vacuo* at 100°C for 30 minutes. Iodine numbers were calculated from the refractive indices of the petroleum-ether-extracted oils determined with a Zeiss dipping-type refractometer, using the regression equation of Zeleny and Coleman (1937). Respiratory activity of the unground samples was determined by the method described by Ramstad and Geddes (1942), which involves measurement of the total carbon dioxide respired after incubation of a suitable weight of the sample for 4 days at 37.8°C (100°F). Certain of the chemical tests were repeated with samples from which all foreign material had been removed prior to grinding.

The results of these studies are summarized in Table II. The rate of respiration of the flaxseed from the fatal bin (No. 407) was over five times that of any of the other samples; this flaxseed had the lowest test weight, the highest percentage of dockage, the highest moisture content, and was of low germinating capacity. The State Seed Testing Laboratory reported only 29.8% of sound seed present in the sample from the fatal bin and 25.4% of seed showing sprout damage or a total purity of 55.2%. The impurities comprised chaff, dirt, broken seed, sprouts, cotyledons, oats, barley, and 4.2% weed seeds (yellow foxtail, green foxtail, wild mustard, Indian mustard, wild rose, wild buckwheat, lady's thumb, Pennsylvania smart weed, pale smart weed, lamb's

quarter, ragweed, large-seeded false flax, barnyard grass). The dry matter composition of the cleaned flaxseed from the fatal bin did not differ greatly from that of the other samples, although it was the lowest in cyanogenetic glucoside content and in iodine value and was the highest in fat acidity. The very high fat acidities of the uncleaned seed from bins No. 407 and 204 are worthy of note; Zeleny and Coleman (1938) and others have pointed out that fat acidity increases with deterioration on storage.

Before the fatal bin was emptied, interseed air samples were taken at 9-foot intervals throughout the entire depth of the bin. This was accomplished by driving ten-foot sections of ¼-inch pipe, connected with couplings, into the flaxseed and withdrawing gas samples by connecting a sample tube and vacuum pump to the upper end of the pipe. The oxygen and carbon dioxide contents of these samples are represented graphically in Figure 1. The composition of the interseed

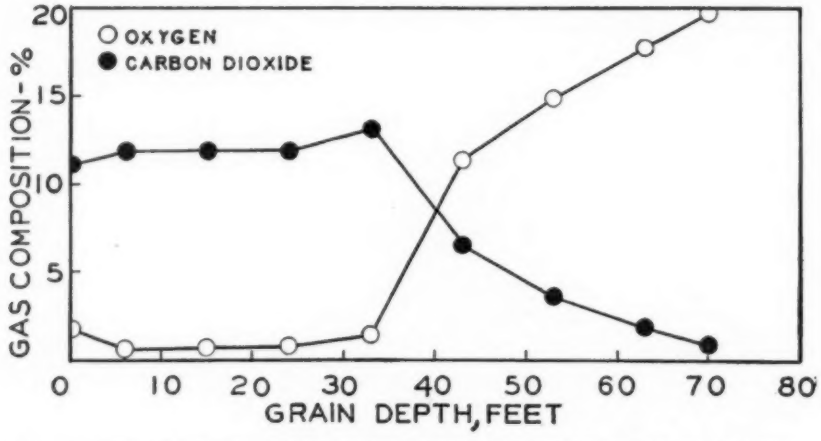

Fig. 1. Oxygen and carbon dioxide content of interseed air at various depths in the fatal bin.

air was relatively constant to a depth of 33 feet, while the samples at successively greater depths showed a progressive increase in oxygen and decrease in carbon dioxide content until at the bottom of the bin, the analysis of the interseed air approached that of normal air.

This result was most surprising and eight samples were taken from the conveyors during various stages in the emptying of the bin in order to ascertain whether variations in the amount of foreign material or in moisture content would serve to explain the differences noted in interseed air. The dockage ranged from 8% to 12%, test weight from 41 to 43 pounds per bushel, and moisture content from 9.1 to 10.5%. These differences do not appear to be sufficiently great to account for the wide differences noted in the composition of the interseed air. It

should be noted, however, that the dockage and moisture contents of these samples were lower than the values given in Table II for the sample taken, of necessity, from the top of the bin. The maximum temperature of the flaxseed observed during the emptying of the bin was 78°F.

TABLE II

COMPARATIVE DATA ON FLAXSEED SAMPLES

	Bin No. 106	Bin No. 204	Bin No. 409	Bin No.[1] 407
Grain and Seed Division, Agricultural Marketing Service				
U. S. Grade	No. 1	Sample	Sample	Sample
Dockage, %	6	10	13	29
Moisture content (Tag-Heppenstall) %	8.2	9.3	9.2	12.4
Weight per bushel, lbs	50	44	43½	32
Minnesota State Seed Testing Laboratory				
Germination, %	88	19	52	35
Chemical analyses of flaxseed[2]				
Moisture, %	7.8	7.9	8.1	9.2
Total nitrogen, %	4.1	4.1	4.0	4.0
Combined HCN, mg HCN per 100 g				
Uncleaned seed	15.6	16.2	16.4	14.7
Cleaned seed	18.8	16.5	18.0	16.1
Oil content, %				
Uncleaned seed	38.8	39.3	36.5	31.2
Cleaned seed	40.4	41.6	40.0	40.4
Iodine value of oil				
Uncleaned seed	171.7	160.4	163.7	139.0
Cleaned seed	176.5	171.1	173.4	170.8
Fat acidity, g KOH per 100 g				
Uncleaned seed	1.03	2.99	1.78	3.22
Cleaned seed	0.19	0.35	0.35	0.37
Respiration, mg CO_2/100 g/24 hr	1.4	5.3	2.5	26.8
Composition of interseed air in bin (six feet below surface)				
Oxygen, %	19.3	8.3	10.1	0.4
Carbon dioxide, %	0.5	9.0	3.3	12.6

[1] Bin in which death occurred.
[2] Analytical results are expressed on a dry-matter basis.

Portions of the samples taken during the emptying of the fatal bin were submitted to the Division of Plant Pathology for examination. The flaxseed was found to be unusually heavily infected with bacteria and fungi and the flora was different from that generally found. Previous examinations made of flaxseed over a number of years indicated that *Alternaria* was by far the most common organism present although *Fusarium*, *Helminthosporium* and *Colletotrichum* were frequently found. The predominating fungi in the current samples, however, were species of *Aspergillus*, *Penicillium*, and *Chaetomium*, whereas *Alternarium*, *Helminthosporium* and *Fusarium* were rare. Other fungi present were species of *Mucor*, *Rhizopus*, *Sordaria*, *Monilia*, and three unidentified. In general, the fungi were of a saprophytic

type. No attempt was made to identify the bacteria but at least five species were represented, none of which was capable of growing under anaerobic conditions.

Discussion

The lethal atmosphere in the fatal bin was the combined result of several factors favoring a low oxygen and high carbon dioxide content. Sound flaxseed has been shown to possess a higher inherent rate of respiration than the cereal grains. The flaxseed in question was above average moisture content, was of sample grade as a result of sprout damage, and contained a high percentage of dockage. These conditions are conducive to high respiratory rates. Moreover, the seed was of low germinating capacity and it is well known that dead organic material is more easily attacked by microörganisms than viable grain. Bacteria and saprophytic fungi were found in great abundance; the seed had been carried over from the previous crop year so that plenty of opportunity was provided for their multiplication on the favorable substrate. The very high respiration of the flaxseed from the fatal bin was undoubtedly due mainly to the respiratory activity of microorganisms. These conditions, coupled with the fact that the bin was nearly filled and had remained closed for several weeks, favored the development of a lethal atmosphere over the seed.

While the inherent respiration of flaxseed is higher than that of cereal grains, death by asphyxiation has been reported when workmen entered bins containing barley, corn, or oats stored at high moisture contents. Precautions should accordingly always be taken to ascertain whether the atmosphere over stored grain is safe before workmen are permitted to enter. The atmosphere may be conveniently tested by lowering a small animal or a fowl into the bin. If a lethal atmosphere is present the worker should be provided with a supplied-air type of respirator, such as a hosemask, or preferably the tank should be thoroughly ventilated and the atmosphere again tested to insure that the ventilation has been adequate.

It must be emphasized that canister masks are designed to remove certain toxic materials and are obviously of no value in atmospheres where there is insufficient oxygen to sustain life. With reference to ventilation, it must be recalled that carbon dioxide, being heavier than air, tends to form a blanket over the grain and it is necessary to disperse this by circulation of the air over the grain in order to secure efficient ventilation in a reasonably short time. Finally, accidents of this type may be avoided by adopting the rule that under no circumstances should anyone enter a grain tank without wearing a safety-belt and having a second person available on the outside to pull the individual out in case of trouble.

The variations in the composition of the interseed air at different depths in the bin indicate that some aeration is taking place even in closed bins; it is logical to anticipate considerable movement of the interseed air as a result of convection currents set up by variations in the temperature of the grain in different portions of the bin. Evidence that ventilation occurs in the bulk storage of soybeans has recently been obtained by Ramstad and Geddes (1942). These workers also reported the presence of traces of carbon monoxide in the interseed air of soybeans undergoing heating but no studies were undertaken on the mechanism of its production.

Summary

Respiratory activity of 4700 bushels of slightly heating, sample grade flaxseed containing about 9.0% moisture upon storage in an interstice, closed, grain-elevator bin filled to within 7 feet of the top for 58 days resulted in the death of an elevator employee. Upon entering the bin, the employee collapsed and although removed within about 10 minutes and given prompt oxygen treatment, death resulted 44 hours later.

The air immediately above the flaxseed contained 1.8% oxygen and 11.1% carbon dioxide, while a sample drawn six feet within the flaxseed contained 0.4% oxygen and 12.6% carbon dioxide. Interseed air composition was uniformly low in oxygen and high in carbon dioxide content to a depth of about 33 feet in the grain; at greater depths there was a progressive increase in oxygen and decrease in carbon dioxide. A sample of interseed air collected a few inches from the bottom of the bin contained 19.8% oxygen and 0.7% carbon dioxide. No free hydrogen cyanide was found but traces of carbon monoxide (0.02%–0.04%) were detected in an independent investigation conducted by the Minnesota Department of Health.

Flaxseed from the top of the bin had a test weight of 32 pounds per bushel and contained 29% dockage. The sample contained 25.4% flaxseed showing sprout damage and gave only 35% germination. Respiratory activity of the uncleaned sample was 5 to 20 times greater and the fat acidity considerably higher than that of other flaxseed undergoing storage in the same elevator. Oil content was similar but oil iodine value and combined hydrogen cyanide were somewhat lower in the cleaned seed from the fatal bin, as compared with other samples.

Bacteria and saprophytic fungi were found in abundance and are believed to be responsible for the abnormally high respiratory activity of the seed from the fatal bin.

Precautions are outlined for preventing the accidental asphyxiation of workmen entering grain tanks.

Acknowledgments

The authors gratefully acknowledge the interest and coöperation of a number of individuals and organizations in connection with various phases of this investigation. The Minneapolis Fire Department provided the services of a rescue squad to assist in the collection of gas samples, the Minneapolis office of the Grain and Seed Division determined the grades reported and the Minnesota State Seed Testing Laboratory made the germination tests. The authors are also indebted to J. J. Christensen, Division of Plant Pathology, who carried out the mycological examinations, to J. A. Schricker, Assistant Chemist, U. S. Bureau of Plant Industry, for determinations of oil content and iodine value, and to P. E. Ramstad, Research Assistant, Division of Agricultural Biochemistry, who determined the respiratory activity of several samples. They also desire to thank L. W. Foker, M.D., Director, Division of Industrial Health, Minnesota Department of Health, for his coöperation in exchanging data and for valuable suggestions in connection with the preparation of the manuscript.

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THE MANGANESE CONTENT OF BREAD AND WHEAT PRODUCTS

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(Received for publication October 5, 1942)

Commercially it is often important to know the degree of extraction of the flour from which a loaf of bread is made. Crumb color and thiamin and iron contents are indicative to some extent. In an enriched loaf, however, since in most cases thiamin and iron have been added, some other factor in addition to crumb color is necessary if the ash of the flour is to be reasonably well estimated. Experiments in this laboratory show that the manganese content of a loaf of bread serves as an indicator of the degree of extraction of the flour used in

making the loaf. The fact that manganese varies with the ash of a flour has been known for some time; for example Albizzati (1936) studied the manganese content of flour in the Argentine and found a definite relationship of manganese to ash.

The manganese content of bread is also of interest from the nutritional point of view. It is thought that our diets are adequate in

TABLE I

THE RELATIONSHIP OF MANGANESE CONTENT OF BREAD TO THE FLOUR USED IN ITS PREPARATION

Manganese per pound of bread (38% mb)	Estimated ash content of flour (13.5% mb)
mg	%
0.9 or less	Under 0.40
0.9 to 1.25	0.40 to 0.44
1.25 to 1.9	0.44 to 0.50
1.9 to 2.3	0.50 to 0.60
2.3 or more	Over 0.60

TABLE II

MANGANESE CONTENT OF WHEAT AND WHEAT PRODUCTS

	Ash	Present study	Literature	
	%	μg/g	μg/g	
Wheats:	—	—	49.0[1]	
Southwestern 1941	1.289	20.0	—	
Southwestern 1939	1.822	43.3	—	
Southwestern 1939	1.601	26.7	—	
Southwestern 1939	1.920	46.6	—	
Northwestern 1941	1.783	36.7	—	
Pacific Coast 1941	1.992	40.0	—	
Pacific Coast 1941	1.707	36.7	—	
Wheat products:				
Bran, 10% moisture	—	—	126.0[1]	
Southwestern	5.760	104.0	—	
Northwestern	6.430	139.0	—	
Red Dog, 11% moisture	—	—	—	
Southwestern	3.030	77.0	—	
Northwestern	2.900	55.0	—	
Germ, 11% moisture	—	—	—	
Southwestern	4.285	102.0	—	
Northwestern	4.320	82.0	—	
Clears, 13% moisture	0.80	—	11.4[2]	
Northwestern	0.722	8.5	—	
Northwestern	1.312	18.2	—	
Southwestern	0.944	9.1	—	
Patents, 13.5% moisture	—	—	3.9[3]	
			Ash	Mn[2]
Southwestern	0.433	3.9	—	—
Southwestern	0.450	4.1	0.40	4.1
Northwestern	0.482	6.1	0.44	5.3
Northwestern	0.438	3.9	0.50	6.3
Northwestern	0.391	3.1	—	—

[1] Skinner and Peterson (1928).
[2] Albizzati (1936).
[3] Peterson and Skinner (1931).

manganese since the daily requirement is probably low, but here again, as with other nutritional factors, the average American diet may be suboptimum. Peterson and Skinner (1931) state that of all classes of foods, the cereal grains contribute most to the manganese supply. McCarrison (1927) pointed out that the milling of cereals reduces the manganese in the diet below the safety point and favored the use of whole wheat bread by children because of its higher manganese content. Recently it has been shown that manganese is important in bone development, and Combs, Norris, and Heuser (1942) have suggested that a condition known as "slipped epiphyses" in children may be due at least in part to a manganese deficiency.

As is shown in Tables I and II, the manganese content of flour is closely related to the ash. Our results suggest that this relationship holds for flours from different geographical areas as well as different years. Based on flour and bread analyses, the table given below is suggested as a guide to the ash of a flour from which a sample of bread was made. As far as our tests indicate, ingredients other than the flour contribute no significant quantities of manganese to the loaf.

Method and Results

Manganese was determined colorimetrically with a spectrophotometer at 535-mμ wave length. The relationship of percentage of transmission to quantity of manganese is in excellent agreement with the Beers-Lambeth law. The spectrophotometer was calibrated with solutions prepared from reagent-grade potassium permanganate. Sufficient flour was taken, for example, to give about 400 mg of ash. The ash was treated with concentrated nitric acid and heated gently to dissolve it; the contents of the crucible were then washed into a 100-ml volumetric flask, the solution was made up to volume and filtered. The first portion of the filtrate was discarded. Fifty ml of the solution was placed in a 100-ml flask, 5 ml of concentrated nitric acid and 0.3 g of potassium periodate were added, and the solution was boiled gently for about a half hour or until the maximum color developed. The solution was cooled, made up to volume, and the transmission determined. The standards were treated in exactly the same way with both the acid and the periodate. The method of treating the ash and the development of the permanganate color were essentially those described by the Association of Official Agricultural Chemists. In the case of bread and products containing chlorides, the chlorides must be removed by adding 5 ml of concentrated sulfuric acid to the 50-ml portion of filtrate and heating until white fumes appear.

The manganese contents of various parts of a mill stream, as related to other nutritional factors, are of interest. Within reasonable

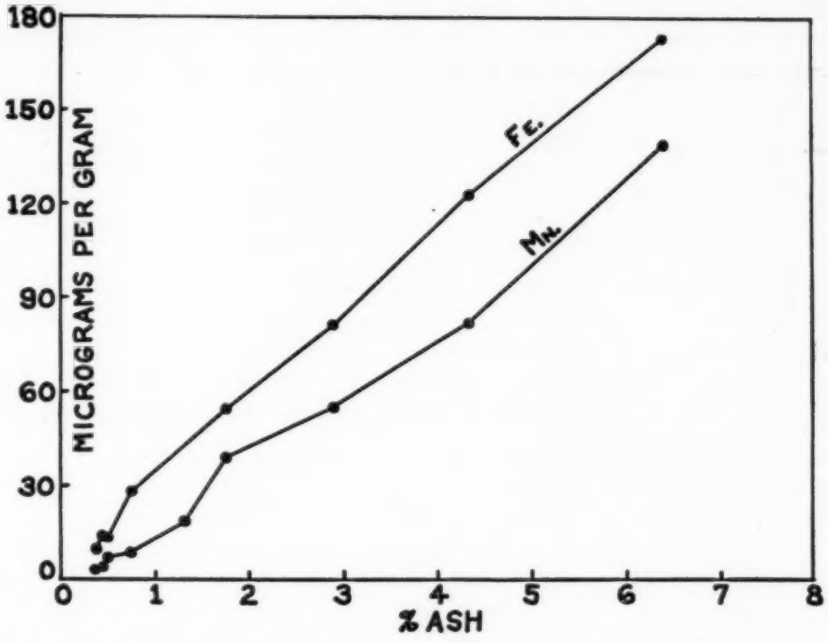

Fig. 1. Relationship of manganese and iron to the ash content of a Northwestern mill stream.

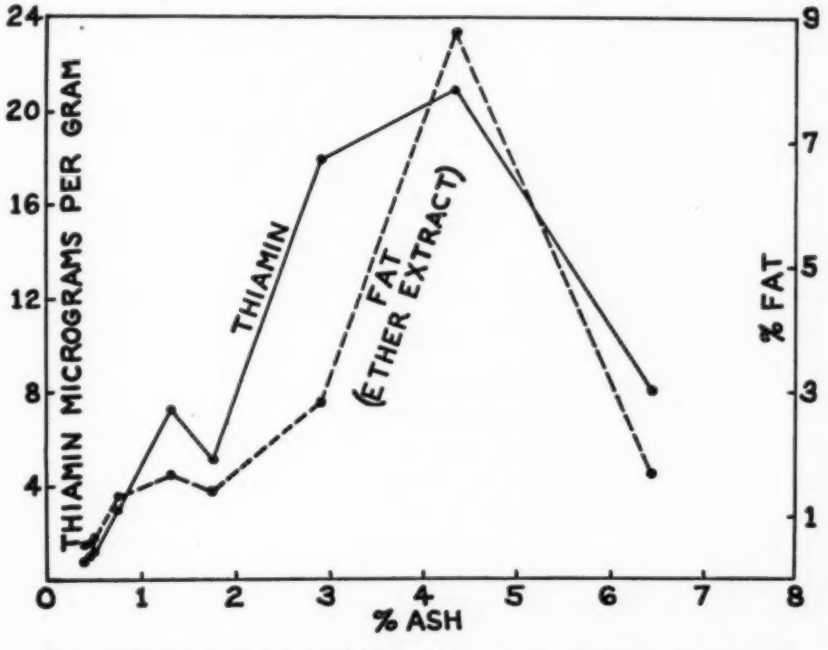

Fig. 2. Relationship of thiamin and fat to ash content of a Northwestern mill stream.

TABLE III

RECOVERY TESTS

	Mn present in flour sample	Mn added	Calculated total	Mn found	Recovery
	mg	*mg*	*mg*	*mg*	%
Whole wheat flour	0.23	0.23	0.46	0.45	98
Short patent	0.144	0.20	0.344	0.350	102

limits the manganese and iron are proportional to the ash. The relationship is more clear-cut in the higher-ash products. Thiamin has a definite relationship to the ash in flours, but as is well known this relationship does not hold throughout the mill stream. It is interesting to note that the ether extract bears a closer relationship to thiamin throughout the mill stream than any other factor yet found. It is at least a theoretical possibility that the cellular mechanisms involved in the production of lipids and of thiamin are closely related.

In the milling of patent flour, the reduction of manganese in whole wheat is somewhat more drastic than reduction of thiamin or iron. The relative physiological availabilities, however, of manganese in patent flour and in the branny portions of wheat are unknown. The difference in manganese content between white bread and whole wheat bread is illustrated in Table IV. The breads were taken from different manufacturers and from widely separated geographical areas.

TABLE IV

MANGANESE CONTENT OF TYPICAL WHITE AND WHOLE WHEAT BREADS

	Mn per pound, 38% moisture basis
	mg
White breads	1.1, 1.5, 1.0, 1.2, 1.1, 1.2, 1.0
Whole wheat breads	10.6, 11.1, 10.5

Summary

A study of the manganese contents of wheat, flour, and bread has been made. Manganese and ash are closely related factors. A manganese determination on enriched bread is often useful in judging the type and grade of flour used in its manufacture. Whole wheat flours average 35–40 μg of manganese per gram, whereas standard patent flours average about 4 μg per gram. These differences are reflected in the manganese contents of white and whole-wheat breads. Whole-wheat bread contains about 10 times as much manganese as white bread.

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THE EFFECT OF SPROUT DAMAGE ON THE QUALITY OF DURUM WHEAT, SEMOLINA AND MACARONI [1]

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Little information is available in regard to the effect of sprouting upon the macaroni-making quality of durum wheat. This problem has received scant attention, in spite of its importance to durum wheat growers, as well as to semolina millers and macaroni manufacturers. Added emphasis was given to the problem by the very wet harvest season of 1941 in the durum-growing region when harvested wheat was exposed for several weeks to weather conditions favorable for sprouting. This resulted in an unusual amount of sprout-damaged durum wheat.

The following investigation was undertaken to study the effects of degree and amount of sprouting upon milling and macaroni quality. A single sample of sound durum wheat sprouted under controlled conditions was used instead of wheat sprouted in the field, because of the diverse environmental conditions to which such wheat would be exposed. Fungus or bacterial infections incident to uncontrolled sprouting might easily be more injurious to quality than the sprouting itself. A further advantage in experimental sprouting is the possibility of having a nonsprouted control as a check.

A suitable quantity of sound amber durum wheat was used for the experiment and germinated under approximately constant conditions. Blends differing as to degree and amount of sprouting were made with the sound wheat on the basis of percentage by weight of the total blend. The percentages were chosen to yield as much information as possible

[1] Joint contribution from the Department of Cereal Technology, North Dakota Agricultural Experiment Station, and the Division of Cereal Crops and Diseases, Bureau of Plant Industry, U. S. Department of Agriculture.
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upon the influence of sprouting upon wheat quality. The blended samples were milled and the resultant semolina processed into macaroni for a comparison of quality characteristics.

Material and Methods

A sound, undamaged sample of commercial Mindum wheat weighing 62.0 pounds per bushel and grading No. 1 Hard Amber Durum was used for this study. It had a bright amber berry, a protein content of 12.1%, and was not sprouted nor weathered. There was no external evidence of fungus infection.

For the purposes of this experiment, the degrees of sprouting were classified into three divisions or stages based on the lengths of plumule and were as follows: (1) The emerging plumule less than half the length of the kernel; (2) the plumule more than half but less than the length of

Fig. 1. Sprouted durum kernels illustrating the three stages of germination: 1st stage, plumule less than half the kernel length; 2nd stage, plumule more than half but less than kernel length; and 3rd stage, plumule longer than the kernel. (Rootlets trimmed short to prevent confusion with plumule.)

the kernel; and (3) the plumule longer than the kernel. The length of the roots was disregarded. The three stages of sprouting are illustrated in Figure 1.

Preliminary experiments were carried out to establish an optimum procedure which would give maximum uniformity of germination and permit a comparison of the effects of different degrees of sprout damage. Factors found to be important in obtaining uniformity were presoaking and chilling, removal of excess carbon dioxide, and the maintenance of uniform moisture content during germination.

The advantage of soaking the wheat before germination may be due to the uneven rate of water absorption by different kernels. Submerging the kernels in water excludes air and retards germination until all kernels have absorbed sufficient moisture for sprouting.

Percival (1921) found that wheat does not germinate until the moisture content has reached at least 30%, which was attained at the end of approximately 10 hours. Leach (1942) followed the rate of water absorption of wheat in contact with water. Water was absorbed very rapidly in the first few hours. The rate fell off gradually with time. Sherwood and Bailey (1926) found that wheat required four or five hours of submergence before it began to germinate.

In the present preliminary studies, the durum wheat was soaked for periods of 3, 8, 10, 12, 16, and 24 hours, and the effects on uniformity of germination were observed. Uniformity of any lot was determined by counting the number of seeds in various stages of sprouting. The greatest uniformity was obtained by soaking 10 to 12 hours. The growth of fungus on the germinating wheat varied inversely with the soaking time, ranging from 30% of obviously infected kernels in lots soaked 3 hours to 5% in lots soaked 24 hours, while lots not soaked were 100% infected. Fungus appeared on 10% to 15% of the kernels in lots soaked 10 to 12 hours, but was not severe enough to cause difficulty, and, therefore, 10 to 12 hours was chosen as the optimum period of soaking.

Miss Edith C. Higgins, Seed Analyst of the North Dakota State Seed Department, suggested chilling the seed before germination to reduce fungus infection and to improve germination. Accordingly, in these preliminary investigations, lots were chilled at 5° to 10°C for periods of 1, 2, 3, 4, and 8 days. It was found that chilled wheat germinated more rapidly than unchilled and the uniformity was improved. Little time was lost by chilling, owing to the increased germination rate. The optimum chilling time appeared to be 3 to 4 days. Less time than this showed little improvement in uniformity, while the 8-day period reduced germination.

It was found that surface kernels in unstirred wheat had roots an inch long, while kernels at the center showed little activity. Presumably this was due to smothering of the latter by carbon dioxide released by the accelerated rate of respiration accompanying germination. Leach (1942) pointed out that within 5 hours after being brought into contact with water the respiration rate in germinating wheat kernels increased to approximately 1,000 times the rate of normal air-dry grain; also, that the respiration rate of germinating wheat continued to increase, though at a more moderate pace. Sherwood and Bailey (1926) carried off the excess carbon dioxide by attaching

an aspirator to the outlet pipe of the germination tank. In the present work occasional stirring of the germinating wheat gave satisfactory uniformity.

Moisture was another variable which affected uniformity of germination. Moist cheesecloth was used above and below the wheat layer to maintain a humid atmosphere. Suitable moisture conditions were maintained by stirring and by adding water as required during chilling and germination.

It was also found desirable to control temperature during germination. Too much time was consumed at lower germination temperatures and fungus infection developed at higher temperatures. The optimum temperature for germination appeared to be 15° to 20°C.

After the desired stage of germination had been reached, the wheat was spread out in a thin layer to dry on a table. Drying was accelerated by two small electric fans. The germinated wheat was dried 24 hours at 25°C at room humidity and stored in bags at a relatively low temperature until milled.

In the procedure finally adopted the samples were treated as follows: (1) Soaked 10 to 12 hours at 15° to 20°C, (2) chilled 3½ days at 5° to 10°C, (3) germinated at 15° to 20°C until the desired stages were reached, (4) during chilling and germination the wheat was stirred thoroughly 4 times during each 24-hour period, and (5) dried 24 hours at 25°C.

The germinator consisted of two trays set end-to-end in a large flat copper tank. The trays were each 2 feet wide, 5 feet long, and 3 inches deep. They were constructed of wooden frames with 16-mesh galvanized screen bottoms. The wheat was soaked, chilled, and germinated in these trays. A double layer of cheesecloth was laid over the bottom of the tray, the wheat spread to a uniform depth of nearly one inch, and the cheesecloth folded back over the top, the ends being allowed to hang over to draw up water by capillary attraction and keep the wheat moist. Water in the tank was maintained at a depth of approximately two inches. During soaking, the trays rested on the bottom of the tank so the wheat was submerged, and while germination was proceeding it was raised slightly above the surface of the water.

To secure the required quantity of wheat, five lots of about 10 pounds each were run. As each of the desired stages of germination was reached, a portion of the lot was removed and dried, the amount removed being roughly proportional to the total amount required at that stage. The rest of each lot was allowed to continue germinating until the next stage was reached. One control lot was soaked and chilled to secure an unsprouted check.

The degree of uniformity of germination attained was determined

by taking a random sample of from 100 to 200 kernels from each lot as it was removed to dry, and classifying the kernels into four groups designated as 0, 1, 2, and 3, these groups corresponding to the stages of sprouting described. Thus kernels in group 0 showed no germination activity, while kernels in group 1 had just begun to germinate, but the plumule was less than half the length of the kernel, etc. The maximum length of the plumule was probably not greater than twice the kernel length. Table I shows the percentages of kernels in each lot that fell

TABLE I

CLASSIFICATION OF SPROUTED KERNELS SHOWING THE DEGREE OF OVERLAPPING IN EACH STAGE
(Modal class indicated in bold type)

Lot	Sprouting stages and percentages of kernels falling into each group [1]											
	First stage				Second stage				Third stage			
	0	1	2	3	0	1	2	3	0	1	2	3
	%	%	%	%	%	%	%	%	%	%	%	%
First	23.2	**76.8**	0.0	0.0	12.0	28.7	**50.9**	8.4	10.7	19.0	14.4	**55.9**
Second	15.1	**84.9**	0.0	0.0	14.7	29.0	**53.3**	3.0	9.2	13.2	16.0	**61.6**
Third	22.5	**77.5**	0.0	0.0	11.5	24.2	**50.0**	14.3	12.0	5.9	5.7	**76.4**
Fourth	22.0	**78.0**	0.0	0.0	14.7	23.5	**57.9**	3.9	10.0	4.1	23.4	**62.5**
Fifth	17.9	**82.1**	0.0	0.0	15.1	6.6	**53.7**	24.6	—	—	—	—
Average	20.1	**79.9**	0.0	0.0	14.6	22.4	**53.2**	10.8	10.5	10.5	14.9	**64.1**

[1] Group for plumule length to correspond with the three stages of sprouting; *i.e.*, Group 0 = percentage with no sprouts. Group 1 = percentage with plumule less than half the length of the kernel. Group 2 = percentage with plumule more than half but less than the length of the kernel. Group 3 = percentage with plumule longer than kernel.

into these four groups and the averages. The averages are not weighted because of the complicating effects of moisture and sprouts. Complete uniformity, of course, was not attained, but the analysis shows that three distinct stages of sprouting were represented, in spite of overlapping.

With the three stages of sprouted durum wheat, a series of blends was made for milling into semolina and processing into macaroni. Each of the three stages was used for a separate series of blends with sound wheat, using 5%, 10%, 20%, 40%, 60%, 80%, and 100% by weight of the sprouted grain. Different stages of sprouting were not blended. Because seed was insufficient the 80% and 100% blends of the third stage and the 100% blend of the second stage were omitted.

The determination of grade was made according to instructions in the *Handbook of Official Grain Standards of the United States* (1939 edition), prepared by the Agricultural Marketing Service, U. S. Department of Agriculture. The milling and processing methods and equipment have been described in detail by Harris and Sibbitt (1942) and will not be discussed in this paper. Analytical procedures were

those outlined in *Cereal Laboratory Methods* (4th edition, 1941).[5] The various blends of sprouted grain caused no unusual difficulties in milling. In processing the macaroni, however, some difficulties in handling were evident. Doughs made from blends high in sprout damage were crumbly and "short," although a dough of normal consistency could be made from them. This apparent "shortness" is probably related to an accumulation of fatty acids following hydrolysis of fat during sprouting. Shattering and checking in the finished product increased with the percentage of damage in the second and third stages of sprouting. Macaroni from the control samples dried satisfactorily.

Discussion of Data

The effects of sprouting upon test weight, percentage of vitreous kernels, grade, and kernel weight are shown in Table II. Soaking and chilling without germination reduced the test weight from 62.0 to 54.0

TABLE II

THE EFFECT OF SPROUTING ON THE TEST WEIGHT, PERCENTAGE OF VITREOUS KERNELS, GRADE AND KERNEL WEIGHT OF DURUM WHEAT

Sample	Test weight	Vitreous kernels	Unofficial grade [1]	Weight per 1000 kernels
	lbs/bu	%		*g*
Control (sound wheat)	62.0	90	1 HAD	36.6
Control, soaked and chilled, no sprouting	54.0	54	4 D	34.4
First stage of sprouting	51.0	0	SGD	34.2
Second stage of sprouting	47.0	0	SGD	33.0
Third stage of sprouting	46.5	0	SGD	31.8

[1] Letters denote first letter of each word in assigned grade, *e.g.*, HAD = Hard Amber Durum.

pounds per bushel. Test weight continued to fall as the degree of sprouting advanced, reaching a low of 46.5 pounds. Even the first stage of sprouting eliminated all vitreous kernels. Kernel weight fell off somewhat with sprouting.

Test weight per bushel, grade, milling, and analytical data obtained from the various blends are shown in Table III. The blends are arranged according to macaroni color score, as shown later in Table IV. Both degree and amount of sprout damage had a marked effect upon weight per bushel and grade, reducing these characteristics from 62.0 to 49.0 pounds and from No. 1 Hard Amber Durum to Sample Grade Durum in the extreme. No definite effect of sprouting upon the protein content of the wheat or semolina was indicated, but semolina yields, both purified and unpurified, were decreased by sprouting. Diastatic activity was greatly affected by the degree and amount of sprout in the blend.

[5] Published by American Association of Cereal Chemists, 110 Experiment Station Hall, University of Nebraska, Lincoln, Nebraska.

TABLE III

COMPARATIVE GRADE, MILLING AND ANALYTICAL DATA FROM THE SPROUTED WHEAT BLENDS

(Results arranged in order of increasing macaroni color score)

Lab. No.	Blend description [1]	Test weight	Unofficial grade [2]	Protein (N ×5.7) [4]		Semolina yield		Ash [4]	Diastatic activity [3,4]
				Wheat	Semolina (purified)	Unpurified	Purified		
		lbs bu		%	%	%	%	%	
41–674	80% 2nd stage	49.0	SGD	12.5	11.1	54.9	37.3	0.59	1155
679	60% 3rd stage	52.0	SGD	12.1	10.7	55.8	35.1	0.59	1150
673	60% 2nd stage	52.0	SGD	12.3	11.3	58.1	41.1	0.64	1022
678	40% 3rd stage	55.5	SGAD	12.2	11.1	59.7	39.1	0.62	1060
677	20% 3rd stage	58.0	SGHAD	12.2	11.2	61.8	41.9	0.61	872
672	40% 2nd stage	56.0	SGAD	12.2	11.3	60.1	40.6	0.62	952
676	10% 3rd stage	60.0	4 HAD	12.1	11.1	61.7	41.2	0.60	717
671	20% 2nd stage	58.0	SGHAD	12.1	11.2	61.9	41.5	0.60	727
675	5% 3rd stage	61.0	3 HAD	12.4	10.5	63.4	42.2	0.61	697
670	10% 2nd stage	60.0	4 HAD	12.1	11.3	61.6	42.4	0.59	596
668	100% 1st stage	51.0	SGD	13.1	11.2	55.3	37.8	0.56	603
667	80% 1st stage	53.0	SGD	12.6	11.1	59.3	40.2	0.61	573
669	5% 2nd stage	61.0	3 HAD	12.4	11.2	62.2	42.2	0.60	515
666	60% 1st stage	55.0	SGD	12.2	11.2	60.6	41.3	0.64	509
665	40% 1st stage	57.0	SGAD	12.2	11.1	61.9	41.5	0.64	403
664	20% 1st stage	59.0	SGHAD	12.2	11.1	62.5	41.1	0.62	321
663	10% 1st stage	60.0	4 HAD	12.0	11.1	62.8	43.0	0.62	254
662	5% 1st stage	61.0	3 HAD	12.2	11.1	58.5	41.8	0.61	247
661	Control, soaked and chilled	54.0	4 D	12.0	11.2	60.1	43.4	0.58	336
660	Control	62.0	1 HAD	12.1	11.2	64.2	42.2	0.62	247

[1] Blends made with sample 41–660 plus indicated percentage of sprouted wheat.
[2] Letters denote first letter of each word in assigned grade, *e.g.*, SGHAD = Sample Grade Hard Amber Durum.
[3] Mg maltose per 10 g semolina.
[4] Calculated to 13.5% moisture basis.

Data obtained from the semolina and macaroni are shown in Table IV. The number of specks in the semolina was not affected by sprout damage, but the absorption was lowered. Color score of the macaroni was closely related to both the degree and amount of sprouting. A high negative correlation was found between visual color score and diastatic activity. In an unknown sample of semolina a high maltose value would probably indicate a low macaroni color score. While the grade was lowered from No. 1 Hard Amber Durum to No. 4 Durum by soaking and chilling the control, the macaroni color score was unchanged.

The effects of the various blends upon the principal quality factors are shown graphically in Figures 2, 3, and 4. Figure 2 shows the effects of the different percentages of each stage of sprouting upon test weight, semolina yield, and semolina ash. Test weight was significantly reduced by the addition of sprouted wheat to the blend, and

TABLE IV

ABSORPTION AND QUALITY RATINGS OF THE SEMOLINA AND MACARONI

Lab. No.	Blend description [1]	Semolina			Visual color score of macaroni (perfect score 10)
		Specks per 10 sq in	Rating	Absorption [2]	
				%	
41–674	80% 2nd stage	20.0	6	25.7	3.5 brownish
679	60% 3rd stage	13.0	4	25.1	3.5 brownish
673	60% 2nd stage	7.0	2	26.0	4.0 brownish yellow
678	40% 3rd stage	10.0	3	25.6	4.0 brownish yellow
677	20% 3rd stage	13.0	4	25.9	4.5 light brownish yellow
672	40% 2nd stage	20.0	6	26.3	5.0 light brownish yellow
676	10% 3rd stage	10.0	3	26.7	5.0 light brownish yellow
671	20% 2nd stage	3.0	1	26.7	5.5
675	5% 3rd stage	10.0	3	26.8	5.5
670	10% 2nd stage	7.0	2	26.8	6.0
668	100% 1st stage	23.0	7	25.8	6.5
667	80% 1st stage	23.0	7	25.6	6.5
669	5% 2nd stage	10.0	3	26.8	6.5
666	60% 1st stage	10.0	3	25.7	7.0
665	40% 1st stage	17.0	5	26.0	7.5
664	20% 1st stage	7.0	2	26.1	8.0
663	10% 1st stage	3.0	1	26.4	8.5
662	5% 1st stage	7.0	2	26.6	9.0
661	Control, soaked and chilled	13.0	4	26.2	9.0
660	Control	7.0	2	26.6	9.0

[1] Blends made with sample 41–660 plus indicated percentage of sprouted wheat.
[2] Calculated to 13.5% moisture basis.

this effect was slightly more marked at the higher percentages of stages 2 and 3. Yield of semolina was not affected by less than approximately 20% of sprouted wheat; more than this, especially of the two last stages of sprouting, decreased the yield. Forty percent of stage 3 reduced the yield nearly as much as 100% of stage 1. No clearly marked trend in the ash was evident, although it appears that a very high percentage of sprouted wheat may tend to decrease this constituent.

Diastatic activity and semolina absorption in relation to sprouting are shown in Figure 3. The first stage of sprouting raised the maltose value from 247 mg to 603 mg, and the second stage increased it to 1155 mg, as compared with 247 for the unsprouted sound wheat. The third stage used in a 60% blend showed a diastatic activity of 1150 mg. The diastatic activity of 100% of the first stage was almost equaled by 10% of the second and was exceeded by 5% of the third. These observations suggest that small proportions of heavy sprout damage may be detected by the high maltose value of the blend. A marked general tendency toward lower absorption with higher concentrations of all stages of sprouting is shown, although the results are not as clearly cut among the blends as was the case with diastatic activity. The range

of absorption covered is not large, but a significant effect is visible in the two last stages of sprouting.

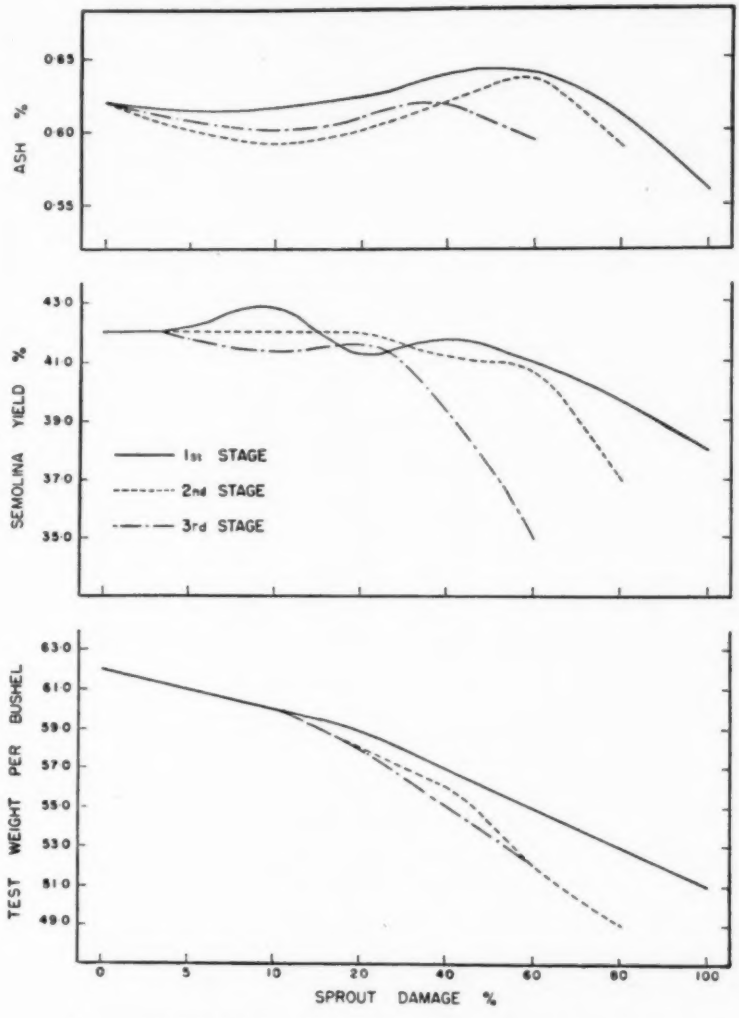

Fig. 2. Effect of different concentrations of the three stages of sprouting upon test weight per bushel, semolina yield, and ash.

Figure 4 shows the influence of sprouting upon the visual color rating of the macaroni. Macaroni color scores decreased consistently with the addition of sprouted wheat. Severely sprouted wheat was especially harmful, and the color scores illustrate its injurious effect upon the commercial value of the products made from it. Five percent of the second stage lowered the color score from 9.0 to 6.5, which is certainly very significant, but an equal proportion of the third stage

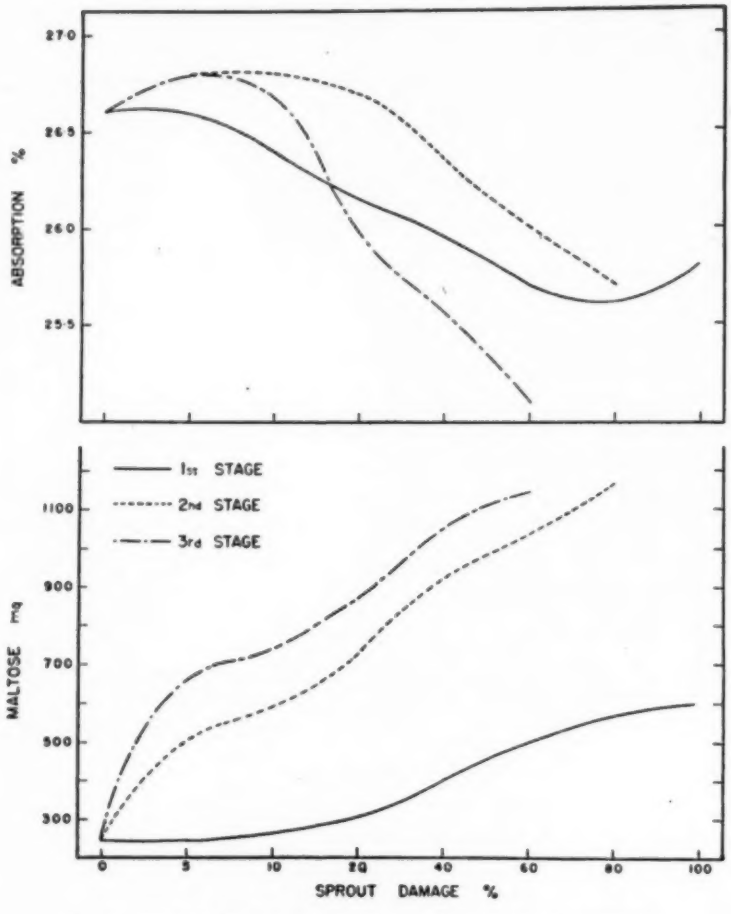

Fig. 3. Effect of different concentrations of the three stages of sprouting upon the maltose figure and absorption.

had a still more drastic effect, rendering the macaroni quite unsuitable for consumption, according to commercial color standards. Lightly sprouted wheat from the first stage was much less injurious to color, but had a noticeable effect when present in 20% or more in the blend. One hundred percent of the first stage produced macaroni which equalled a 5% blend of the second stage, and was superior to 5% concentration of the third stage. These results obtained, compared with those from a study by Harris and Sibbitt (1942), suggest that blights and similar forms of damage are more injurious to durum quality than is sprout damage.

The results of the present study justify the following general statement: Sprouting adversely affects test weight per bushel, grade, semo-

lina yield, and macaroni color. Diastatic activity is greatly stimulated. Of these various effects those connected with test weight, semolina yield, and color are the more important from the standpoint of commercial utilization. Returns to the farmer would be decreased because of lowered bushel weight and grade. If the sprouting is slight,

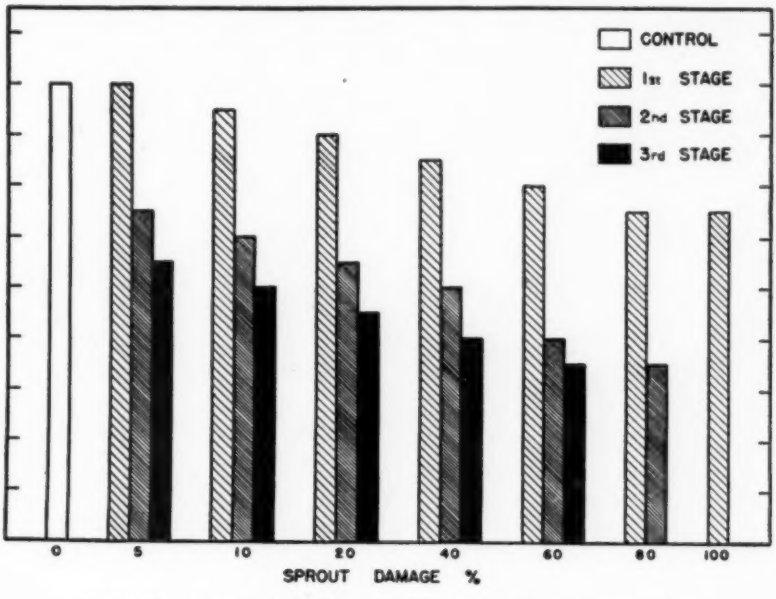

Fig. 4. Visual macaroni color score as influenced by different concentrations of the three stages of sprouting.

say less than one-half the kernel length, and constitutes not more than 10% by weight of the total wheat mix, the effect upon yield, diastatic activity and color is not great, but test weight and grade are still reduced. The influence upon grade is especially marked, causing a decrease from No. 1 Hard Amber Durum to No. 4 Hard Amber Durum.

Summary and Conclusions

Aliquots of a sample of sound hard amber durum wheat were sprouted under approximately uniform conditions for varying lengths of time in order to obtain three distinct stages of sprouting. These "stages" were delimited by the length of sprout obtained. Each of these three stages was then blended in various proportions by weight with the original sound wheat to obtain mixes for experimental milling. These were milled and the resultant semolinas processed into macaroni by standardized methods. Various chemical determinations were made on the material, as well as a visual color score determination of the macaroni.

While the different degrees of sprouting did not affect the ease of milling the blends, an effect was noticeable upon the properties of the dough during macaroni processing. Doughs made from blends containing a high percentage of badly sprouted wheat were crumbly and "short," but after the customary amount of kneading appeared to have normal consistency.

Weight per bushel was consistently lowered by sprouting. Semolina yield was significantly reduced when over 20% of sprouted wheat was included in the blend. Little influence upon semolina ash was noted, except that high percentages of sprouted wheat showed a slight trend toward reduction.

Diastatic activity was greatly influenced, both by the percentage of sprouted wheat in the blend, and by the degree of sprouting, while absorption was generally lowered by the same factors. The effect of sprouting was most marked upon diastatic activity and macaroni color, and in the present instance these two factors bore a high negative relationship to each other. The determination of diastatic activity may be a convenient method for predicting the probable macaroni color of a sample suspected of containing sprout damage, provided no complicating factors such as blight or other damage are involved. Ten percent blends of the second and third stages had more effect upon both diastatic activity and color than 100% of stage 1.

Five percent of heavy damage reduced the color score 40%. The second stage of sprouting decreased the score less severely. The wheat from the first stage had a noticeable effect at a concentration of 20% in the blend. The macaroni had a brownish coloration when made from blends high in severely sprouted wheat.

Soaking and chilling without germination significantly reduced test weight and grade but did not affect macaroni color.

It appears from the data that length of sprout is more important in relation to quality than the percentage of sprouted kernels present.

Acknowledgment

The authors acknowledge the assistance provided by the Work Projects Administration, Research and Records Division, through the operation of Seed Testing Project O.P. 165-1-73-144 during this investigation.

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CONSTANCY OF RANK OF DURUM WHEATS IN MACARONI COLOR

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(Received for publication September 23, 1942)

This paper presents macaroni color differences on fairly constant groups of durum varieties and hybrid strains grown at the Langdon Substation, Langdon, North Dakota, for the years 1929 to 1941, exclusive of 1936.

Color receives more attention than any other macaroni quality characteristic. It is not necessarily more important than such attributes as nutritional value, cooking properties, protein content, flavor, or breaking strength of the macaroni, but it is more likely to be defective in commercial macaroni, and is more easily recognized by the buyer. Color weighs heavily in macaroni sales appeal. Therefore, in any durum wheat improvement program, macaroni color of the varieties and hybrid strains is of paramount importance.

Macaroni color is modified by many factors, some hereditary and some environmental. The influence of hereditary factors is illustrated by the fact that good durum wheats produce brighter and more yellow macaroni than do wheats of other classes. Furthermore, certain varieties of durum are much superior to others. The Mindum variety has become accepted by the trade because it produces macaroni that is clear, bright, and translucent. Contrastingly, Pentad, or "red durum," produces dull, reddish, opaque macaroni.

Examples of environmental factors influencing macaroni color are climate, soil, disease, or even storage conditions and processing technique. The importance of climate and soil is indicated by the fact that virtually all the durum wheat produced in the United States is grown in North Dakota, Minnesota, and South Dakota, with 75% being produced in North Dakota. In years of high rainfall, the color characteristics of the crop are different from those in a dry year. A disease such as black point may cause splotchy macaroni color, or ergot may cause black specks. Stem rust may shrivel the kernels, increasing the amount of yellow color per given weight, but making

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the removal of bran particles more difficult. Finally, the color of the best durum may be ruined by poor storage or careless processing.

Binnington and Geddes (1937) milled and processed into macaroni several durum wheat varieties and hybrids grown at three locations in western Canada in 1934 and 1935. They analyzed the macaroni color of the various samples and calculated "single-score color values" by means of a disk color analyzer. The calculated color values ranked the varieties in identically the same order as a visual classification.

Fifield, Smith, and Hayes (1937), in describing a small-sample technique, reported visual color rankings of macaroni from several durum varieties grown at Langdon, North Dakota, from 1929 to 1935. Ranking of the varieties in different years was similar, despite variations in season. Macaroni color of durum samples grown at Langdon in 1934 and 1936 was analyzed with a Novadel-Agene colorimeter. Percentage of yellow color ranked the varieties in essentially the same order as notes on visual appearance of the macaroni. "Macaroni disks" from small samples of semolina gave disk colorimeter results comparable to the experimental macaroni.

A more thorough study of the factors related to macaroni quality was undertaken by Binnington and Geddes (1939) from grade, bushel weight, semolina yield, carotenoid content, macaroni breaking strength, and tenderness score determinations on eleven durum varieties grown in western Canada in 1935, 1936, and 1937. Only Arnautka, Mindum, and Akrona consistently produced commercially acceptable macaroni. They emphasized that macaroni quality cannot as yet be predicted from any single analytical test applied to the wheat, referring especially to carotenoid content.

Fifield, Clark, Smith, Hayes, Christie, and Hoffecker (1937) reported on macaroni made from durum samples grown at a number of different locations in the Great Plains area for the crop years 1932 to 1936. Harris and Knowles (1940) and Harris and Sibbitt (1942) reported on macaroni made from North Dakota durum samples grown in 1938, 1939, and 1940. Visual notes on the macaroni color ranked the varieties in much the same order as did previous workers, even though season, year, and location were different.

Materials and Methods

In connection with the durum wheat improvement program of the Bureau of Plant Industry, U. S. Department of Agriculture, cooperating with the North Dakota Agricultural Experiment Station, a group of standard durum varieties and new hybrid selections was grown each year at Langdon, North Dakota, in triplicated 1/60-acre plots to com-

pare them for such agronomic characters as rust resistance, strength of straw, earliness, yield, and test weight.

In most years, from eight to twelve durums were included in the trial. Some standard varieties were grown continuously throughout the period, while others were discarded to make room for new hybrid strains as the latter became available.

Each year (excepting 1936, owing to drought), 10-pound samples of each were processed into macaroni in the experimental laboratory of General Mills, Inc., and the samples rated for macaroni color. The samples were identified by number only, while being processed and judged. The experimental equipment will not be described in detail. It was built up over a period of years and is essentially similar to that described by Fifield (1934), Binnington and Geddes (1936), and Harris and Sibbitt (1942).

The macaroni color scorings were based on a visual color score in daily use by the laboratory for durum buying and mill-control operations. A standard commercial semolina was used to maintain a uniform base and the samples were scored in percentage units ranging from "very poor" at 40% to "excellent" at 80%. While these were subjective scorings, they were very satisfactory under the conditions of this study. All scorings were made by the same operator in each of the twelve different years, an operator who was in touch with this work the year round. A disk color analyzer would have the advantage of removing some of the personal factor in these scorings, but none was available. It still would have been necessary to check the results against the visual color scores.

This cooperative project was begun to obtain from a commercial laboratory unbiased evaluations of the macaroni quality of new hybrid strains in comparison with Mindum check samples grown under comparable conditions. As the tests were continued over a period of years, they have taken on added significance, bringing out a consistency in the ranking for macaroni color of varieties continued throughout the period.

Experimental Results

The macaroni color scores each year for the more important durums are tabulated by groups in Table I. Groupings were made on the basis of similarity of macaroni color; *i.e.*, Monad and Golden Ball were invariably similar and produced poor macaroni, and constituted one group; selections of Kubanka were grown for a period of years and made up a second group; a third group contained only Mindum and K-75, which are preferred commercially for macaroni color; and in the fourth group were included new hybrid strains from the breeding pro-

TABLE I

MACARONI COLOR SCORES FOR GROUPS OF DURUM VARIETIES AND HYBRIDS GROWN IN FIELD PLOTS AT LANGDON, NORTH DAKOTA, 1929 TO 1941 (1936 OMITTED OWING TO DROUGHT)

Year grown	Variety or hybrid group	Group No.	Macaroni color scores									Av. group score
			40 VP	45 P-	50 P	55 F-	60 F	65 FG	70 G	75 VG	80 Ex	
1929	Mindum—K-75	III							2			70
	Hybrid strains	IV			3		1					53
1930	Monad—Golden Ball	I			1	1						53
	Kubanka selections	II				1	1	1				60
	Mindum—K-75	III							2			70
1931	Monad—Golden Ball	I	1	1								43
	Kubanka selections	II			1		2	1				59
	Mindum—K-75	III									2	80
	Hybrid strains	IV				2						55
1932	Monad—Golden Ball	I	1	1								43
	Kubanka selections	II				1	1	1				60
	Mindum—K-75	III							1	1		73
	Hybrid strains	IV			1	1						53
1933	Monad—Golden Ball	I	2									40
	Kubanka selections	II						3				65
	Mindum—K-75	III								1	1	78
	Hybrid strains	IV				2						55
1934	Monad—Golden Ball	I	1	1								43
	Kubanka selections	II						2	1			67
	Mindum—K-75	III								2		75
	Hybrid strains	IV			1	1						53
1935	Monad—Golden Ball	I	2									40
	Kubanka selections	II				2	2					58
	Mindum—K-75	III						2				65
	Hybrid strains	IV				2						55
1937	Monad—Golden Ball	I	2									40
	Kubanka selections	II						2				65
	Mindum—K-75	III								1	1	78
	Hybrid strains	IV			1				1			60
1938	Monad—Golden Ball	I	1	1								43
	Kubanka selection	II				1						55
	Mindum—K-75	III							2			70
	Hybrid strains	IV		1	1	1			2			58
1939	Monad—Golden Ball	I		1	1							48
	Kubanka selection	II					1					60
	Mindum	III								1		75
	Hybrid strains	IV								2	1	77
1940	Monad	I	1									40
	Kubanka selection	II				1						55
	Mindum	III					1					60
	Hybrid strains	IV		1	2	3	3					54
1941	Monad	I	1									40
	Kubanka selection	II				1						55
	Mindum	III							1			70
	Hybrid strains	IV						4	1	4	1	71

gram. Groups I, II, and III were relatively constant genetically, while Group IV was variable.

The figures under the various color-score classes show the number of varieties or hybrid strains from the indicated group falling in each class. The last column gives the average score for each group each year. Thus, in 1929, both Mindum and K-75 received color scores of 70, or "good," while three hybrids scored 50 and one 60 with an average for the hybrid group of 53. In 1930, Monad or Golden Ball scored 50 and the other scored 55.

Since some of the durum varieties were not grown continuously throughout the 12-year period, the averages cannot be regarded as highly accurate, but they are believed to be representative for each group. The number of Kubanka selections varied from one to four each year, as older selections were replaced by new ones. Golden Ball was dropped from the trials in 1940 and K-75 in 1939. The hybrid group was especially variable and intentionally so, since those shown to be poor in color or deficient in other respects were dropped and newer strains took their places.

Despite wide seasonal variations in macaroni color, the relative rankings of the first three groups remained unchanged. Group I ranked poorest, the average color scores ranging from 40 to 51; Group III ranked best and Group II ranked intermediate each year.

Although only one location is considered, the seasonal environment ranged from very favorable in some years to very adverse in other years for the production of good-quality durum. In 1937, a favorable year, the bushel weight of Mindum was 63.5 pounds, while in 1935, stem rust reduced Mindum to 46 pounds per bushel. In other years the environment was modified by extremes in drought, heat, grasshoppers, black-point fungus, late seeding, and sprouting in the shock. Under the most adverse conditions, Mindum and K-75 scored better than did Monad or Golden Ball in the most favorable year. The Kubanka selections represented more diverse types, but invariably scored below Mindum and K-75, and generally better than Monad or Golden Ball.

Usually the Kubanka selections were graded down because of lack of yellow color, while Monad and Golden Ball were graded still lower because of dullness and gray or reddish shades.

Color scores for Group IV show the improvement in general level of macaroni color that has been attained by breeding. Thus the various hybrids tested and discarded up to and including 1935 were generally inferior to the Kubankas, and decidedly poorer than Mindum. In 1937 and 1938, some of the newer hybrids approached Mindum in macaroni color, but it was not until 1939 that some were available that rated equal to Mindum. All these better hybrids resulted from a back-

crossing program for improved stem-rust resistance, with Mindum as the recurrent parent. In 1941, ten hybrids were tested, all significantly better than Kubanka, and some were rated above Mindum. The relation between color scores of the representative varieties each year and the progress that has been made by breeding is illustrated in Figure 1.

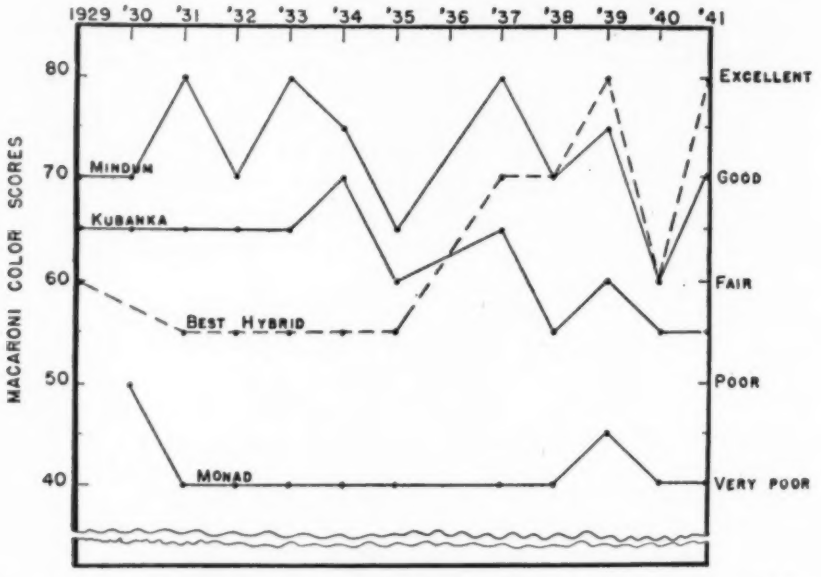

Fig. 1. Macaroni color scores for Mindum, Kubanka, Monad (D-1), and the best-quality new durum hybrid available in the indicated years from field plots grown at Langdon, North Dakota, 1929–41.

This shows the color scores for Mindum, Monad, and the best-colored selection of Kubanka, as compared with the best hybrid in each year. Even though there was considerable variation in the color score for a given variety in different years, the relative position of the varieties one to another was very constant from year to year. The best hybrid in 1935 and in previous years was distinctly inferior to the best Kubanka selection, but the best in 1939, 1940, and 1941 was equal or superior to Mindum.

Similar results were secured with other durum varieties and hybrid strains grown and tested for less than the 12-year period and not reported herein.

The similarity in relative color scores of the different varieties from year to year suggests that it may not be necessary to grow and test new hybrids for many years in order to evaluate them at least roughly. Even a single year's results, in comparison with a suitable check, may have considerable significance. Recognition of finer differences will probably require more time.

Summary

Durum wheats grown in field plots at Langdon Substation, Langdon, North Dakota, from 1929 to 1941, were processed each year into macaroni and scored for macaroni color. Some of the varieties were grown continuously throughout the period while others, especially new hybrid selections, were replaced from year to year with newer material. The varieties were classified into three groups on the basis of macaroni color, and compared with a fourth group, the hybrid strains.

The results of these field and laboratory tests suggest the following conclusions: (1) Ranking of the durum wheat varieties for macaroni color was relatively constant regardless of seasonal fluctuations. (2) New durum hybrid strains bred for improved stem-rust resistance and tested in the later years were much superior in macaroni color to those available for testing in the early years of the period. (3) A new variety or hybrid may be evaluated for macaroni color from a relatively small number of tests, if compared with Mindum or other suitable varieties grown under like conditions and processed comparably.

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THIAMIN LOSSES IN TOASTING BREAD

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(Read at the Annual Meeting, May 1942)

Surveys show that much bread is consumed as toast. In some areas this is said to exceed 35% of the total consumption. Since thiamin is heat-labile and it is known to be partially destroyed in the baking process, some additional destruction would normally be expected during the toasting process. Hoffman, Schweitzer, and Dalby,[2] using assay methods then available, were unable to show any loss of thiamin in white bread even after "heavy" toasting. Since that time methods of thiamin assay have been improved so that smaller amounts of thiamin can be measured.

Enriched bread standards, providing for a minimum and maximum vitamin content, have been proposed and recommended by the Committee on Food and Nutrition of the National Research Council. Enriched bread now on the market is produced in compliance with these standards. It seems desirable that figures be made available showing the thiamin losses occurring during toasting of unenriched white bread, enriched white bread, and 100% whole wheat bread.

Experimental

Enough loaves of each type of bread used were purchased from one delivery to make up all samples. As the loaves were unwrapped the end and next-to-end slices were discarded and the remaining slices distributed into covered glass tobacco jars which were filled to the top by ten slices. By this procedure each sample was made fairly uniform in composition and weight. Experiments showed that, although the jars used were not sealed, no weight was lost in standing longer than the period of the determinations.

The toaster was of the usual household type in which the slices jump up when toasted. The toaster was not new at the time of these tests; hence changes due to a "breaking-in" period were eliminated. To assure fairly constant heat and humidity two slices were toasted and discarded before each sample was taken. Experience showed that the timing device on the toaster was not absolutely constant in action, and so the toaster was set for a period slightly longer than desired in the tests, and each pair of slices was removed by hand at the indicated time. After being toasted each pair of slices was transferred into a

[1] Present address: Hollywood Candy Co., Centralia, Illinois.

[2] Chas. Hoffmann, T. R. Schweitzer, and G. Dalby: Loss of thiamin in bread on baking and toasting, Cereal Chem. 17: 737 (1940).

TABLE I

SAMPLES OF BREAD

	100% whole wheat	Unenriched white	White enriched
Wt. of 10 slices	263.1 g	267.3 g	287.8 g
Thickness of slice	12 mm ($\frac{1}{2}$″)	15 mm ($\frac{5}{8}$″)	16 mm ($\frac{5}{8}$″)
Moisture as rec'd	37.0%	36.6%	38.0%

second glass jar. When the series of ten slices was toasted they were weighed.

After air drying the samples were ground and carefully mixed. Sample bottles were filled to the top and moisture and thiamin were determined on each sample. It was found that in thiamin loss, 5-second increments were not sufficiently large to aid in showing changes. Accordingly data presented here include only the samples in each series toasted 0, 30, 40, 50, 60, and 70 seconds. Ten persons were asked to

TABLE II

THIAMIN AND MOISTURE IN BREAD AND TOAST
(All data converted to 38.0% moisture basis)

Toasted	Moisture	Moisture loss	Thiamin	Thiamin loss	Thiamin in 6 slices toast
seconds	%	%	*μg/g*	%	*mg*
UNENRICHED WHITE BREAD					
0	38.0	0.0	0.86	0.0	0.138
30	32.7	14.0	0.78	9.2	0.126
40	31.3	17.6	0.67	22.1	0.108
50	29.2	23.1	0.69	19.7	0.110
60	24.3	36.0	0.63	26.7	0.102
70	23.0	39.5	0.59	31.4	0.096
ENRICHED WHITE BREAD					
0	38.0	0.0	2.76	0.0	0.496
30	34.6	9.0	2.62	5.2	0.454
40	30.9	18.6	2.57	7.0	0.444
50	29.9	21.4	2.40	13.0	0.414
60	28.4	25.3	2.35	15.0	0.406
70	26.2	31.0	2.29	17.0	0.396
100% WHOLE WHEAT BREAD					
0	38.0	0.0	3.36	0.0	0.532
30	32.1	15.6	3.22	4.0	0.506
40	30.0	21.0	3.08	8.2	0.486
50	29.9	21.3	2.94	12.5	0.462
60	25.6	32.6	2.85	15.3	0.450
70	24.5	35.5	2.65	21.0	0.418

view the toast samples and express their preference as to degree of toasting. Eight preferred samples toasted 50 seconds; one chose 40 seconds, and the tenth said he liked his toast between the 40- or 50-second samples. Table I gives data describing the bread samples used.

Thiamin assays were made by the fermentation method of Schultz, Atkin, and Frey.[3] Because of the small differences expected between the samples, many were assayed in quadruplicate. All results are the means of duplicate or quadruplicate assays, which agreed within 5%. In assaying low-potency samples such as the unenriched white bread toast, there are two methods of procedure: one may use an aliquot sample sufficiently large to provide 2 μg to 4 μg of thiamin (for the fermentometer used). This method is open to the possible objection that the large amount of starch and other suspended matter introduced into the fermentometer may have some effect on the fermentation. Or one may use a constant amount of sample (*i.e.*, 1 g) and to each fermentometer add sufficient of the thiamin standard to make certain the total amount present is within the range of the test. Although the thiamin activity due to the sample is a small part of the total thiamin activity and possible errors in dispensing the standard thiamin will be magnified in the assay results, the latter method was chosen for this study. Table II gives the results of the thiamin assays as applied to the different samples of toast.

Discussion

From the data in Table II it appears that the destruction of thiamin in toast is due to the degree of penetration of heat into the slice as well as to the length of time the toasting process continues. The amount of thiamin destroyed in all three types of toast is related to the degree of toasting. It was first thought that there might be a relationship between the loss in moisture during the toasting period and the loss of thiamin. However, closer examination of the data shows that while these two losses are roughly parallel they are not mathematically related by any simple equation.

The apparent percentage loss of thiamin in the toast made from unenriched white bread is higher than thiamin losses from either the enriched white bread or the 100% whole wheat bread. However, this may well be a discrepancy introduced by the assay method used and the difficulty of measuring accurately such a small concentration of thiamin as remained in this toast. Hoffman and co-workers in 1940 were unable to show any difference in thiamin content between the toast and the control in this range of values.

[3] A. S. Schultz, L. Atkin, and C. M. Frey: Determination of vitamin B^1 by yeast fermentation method, Ind. Eng. Chem. (Anal. Ed.) **14**: 35–39.

Summary

Three types of bread on the Chicago market were assayed for thiamin by the fermentation method. Toast was prepared from each type of bread by toasting portions of the bread for five different intervals of time from 30 to 70 seconds. Data are presented showing the moisture content of the bread and toast, the thiamin content, and percentage of loss in the toast and the amount of thiamin available in 6 slices of the toast.

AN AUTOMATIC GAS RECORDING APPARATUS [1]

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(Received for publication October 7, 1942)

The gassing properties of flour have long been considered of great importance in bread making and a knowledge of these properties is valuable in appraising flour quality. In quality test baking by the formula suggested by Larmour, Geddes, and Whiteside (1933), as used in this laboratory on experimentally milled flours, it is assumed that a deficiency in gassing power may exist. In normal crop years this has been especially true when the flours were experimentally milled from the higher grades of Canadian wheat. This, however, may not always be the case, as has been pointed out by Elion (1932), who stated that "important flour properties may be obscured if sugar is added to every baking test," and also by Collatz and Racke (1925), who have indicated that care should be taken in adding diastatic malt to all flours. These findings indicate a weakness in our present system of test baking which the present work is an endeavour to overcome.

Methods of measuring the gassing value of a flour, in contrast to diastatic activity, are purely physical, the results representing the amount of gas evolved during fermentation. One of the earliest workers to recognize the importance of gassing properties in bread making was Jago (1895) who in his publication devoted a paragraph to the discussion of enzymes and diastase and showed a simple gasometer.

Following work observed at the Research Association of British Flour Millers at St. Albans, England, in 1933, an apparatus employing the principle of Kunis, as shown by Elion (1933), was constructed in the laboratory of the Cereal Division. This apparatus was found to have many weaknesses and, like the apparatus of Markley and Bailey

[1] Contribution No. 125 of the Cereal Division, Dominion Experimental Farm, Ottawa, Canada.

(1932), required almost constant attention as did the simple gasometer of the Jago type suggested by Bailey (1939). The straight manometric apparatus of Sandstedt and Blish (1934) has emerged as probably the simplest of the earlier routine methods although the apparatus of Malloch (1939) has many desirable features. The Fermentograph by Brabender (1934) was an improvement from a recording standpoint but was open to error as shown by Schmalz and Sullivan (1938). With no satisfactory apparatus or test available which automatically recorded in units of time the rate of gas evolved, construction of the apparatus described below was commenced in 1939.

The apparatus, essentially a recording manometer, consists of: (1) the mechanical portion of the apparatus, (2) a water bath to maintain

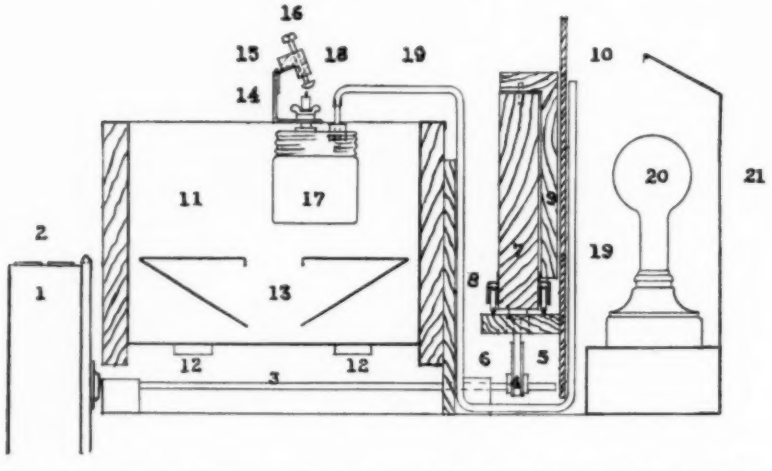

Fig. 1. Apparatus that provides an automatic record of the rate of evolution of gas by doughs.

the temperature of the dough in the manometric system, (3) a carrier on which is transported the recording paper, (4) a series of manometric units, (5) a bank of lights to register the heights of the liquid columns in the manometer tubes, and (6) a shield between the recording paper and the lights, so cut that only a narrow slit allows light to pass through the manometer tube and register on the recording paper.

The mechanical portion as shown in Figure 1 consists of a train of gears which operate a rotary switch. This rotary switch (1) is bolted to the final wheel of the train of gears. On this switch are mounted two electrical contacts (2), one of which operates the lights and one of which operates a magnetic closure. The axle (3) of the final wheel of the gear train is extended under the water bath to the paper carrier. Under the latter an eccentric (4) is fitted to a pitman, the front of which carries a pinion (5) which operates against a ratchet (6) on the under-

side of a movable carriage (7). This carriage (7) rolls on a track (8) and for convenience the recording paper is affixed to a paper holder (9) which is lighter and more easily removed from the machine than is the carrier. The shield (10) protects the recording paper from the light source except for slits which are cut vertically in the shield behind each tube.

The water bath (11) is thermostatically controlled. It is heated by two strip heaters (12) closely applied to the bottom of the bath. Inside the bath and above the strip heaters is a false bottom (13) the underside of which is sloped to throw the rising hotter water outwards towards the sides of the tank. The top of the false bottom is open down the center of its length to permit a return of cooling water to the bottom of the bath on the principle of convection. Lengthwise of the bath and attached to the ends is an L-shaped bar (14). This bar supports the jar (17) by a winged nut on the top valve (18). To the vertical side of the L bar is hinged a bracket (15), which is fitted with adjustable pawls (16) to bear against the pneumatic valves (18). These pawls are brought down on top of the pneumatic valves by the magnetic closure, the built-up pressure is released, and pressure inside the manometric system returns to atmosphere.

The manometric units consist of a container with a screw-on lid (17). The lid is fitted with a pneumatic valve (18) and a hole into which the one end of a manometric tube, partly filled with opaque liquid, is tightly fitted. A bank of lights (20) with a reflecting shield (21) provides illumination for marking the blueprint paper.

In practice, a strip of blueprint paper is attached to the paper holder (9), which is placed on carriage (7) behind the shield (19). The test dough is placed in the jar (17), which is attached to the L bar (14) by a winged nut on the pneumatic valve (18). The manometric tube is connected to the jar and the apparatus started. The train gear turns the rotary switch once in a predetermined time. The eccentric (4), being attached to the same axle (3) as the rotary switch, will turn once in the same time. The eccentric actuates the pawl (5) which engages behind a tooth on the ratchet (6) and moves the carrier forward an equal distance with each revolution. As the switch (1) rotates, one contact closes, lighting the bank of lights (20). These remain on so long as the contact is closed, producing on the blueprint paper behind the slit on the shield (10) and above the liquid in the manometric tube (19) a mark, the lower end of which indicates the height of the column of the opaque liquid in the tube. A cut through the shield in a horizontal position at the same height as the liquid at atmospheric pressure allows the light to pass through the shield and marks the base from which the height of column is measured. Repetition of pressure build-

ups and exposures produces a graph over a period of time of the type shown in Figures 2, 3, and 4.

The formula used in making test doughs was as follows:

Flour	20 g	100%
Yeast	0.6 g	3%
Salt	0.24 g	1.2%
Water	10 ml	50%

Doughs were mixed by hand. By preliminary investigation a 20-g sample was shown to be the size best suited for this size of apparatus. The 3% yeast rate was chosen because it gave a sharp end point, as is shown in Figure 2. Salt was added to obtain a more normal initial curve, the need of it being shown in Figure 3. The absorption is kept to a minimum to retard the swelling of the dough during fermentation. The initial curve, the curve given by a flour with the above formula, shown in Figure 4, does not correspond with those of Larmour and Brockington (1934) but closely follows the general outline of those published by Eisenberg (1940), having a primary and secondary peak.

While no special investigations have been made on the subject of the lag in fermentation which causes this double peak, it appears to be closely associated with the supply of, and demand on, sugars within the dough. This agrees with the views of Amos (1941), whose findings tend to show that the use of the total gas figure to indicate high gassing properties may be erroneous as a result of factors influencing the rate of gas production before or after the peak is reached. From our own work, confirming the findings of Larmour (1934) and others, it has been

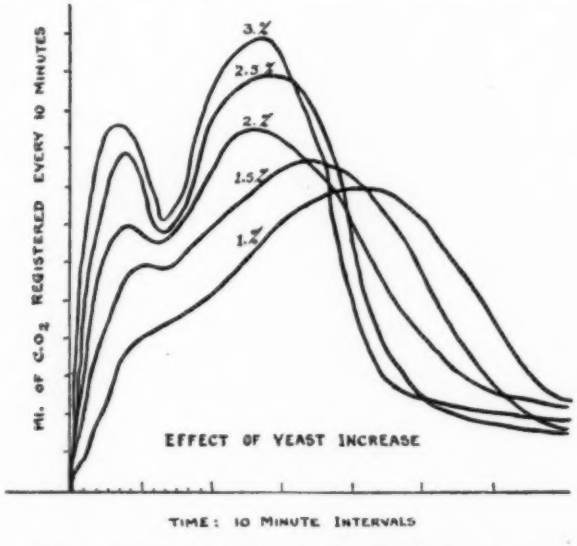

Fig. 2. The effects of increases in yeast on production of gas.

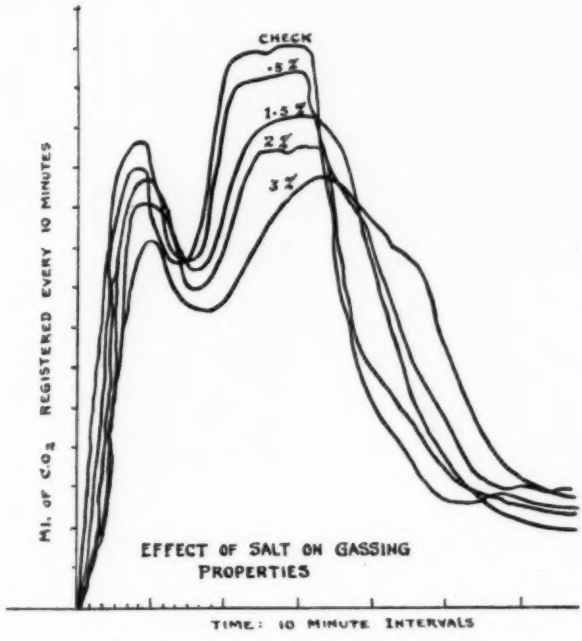

Fig. 3. The effect of salt on the production of gas.

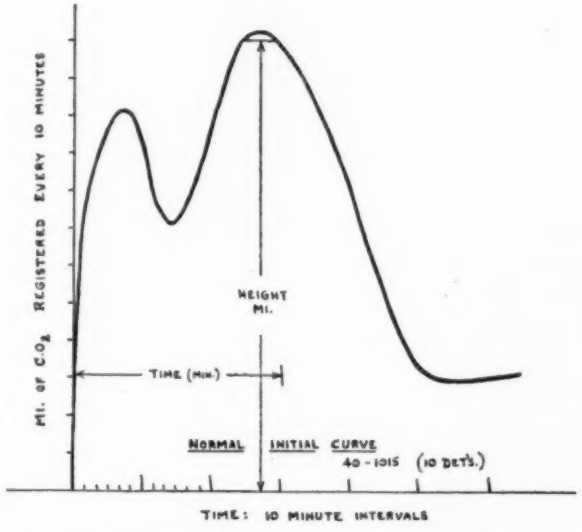

Fig. 4. A normal initial gas-production curve, showing method of measuring time and height factors for calculating the gassing figure.

shown that the need for high gassing properties is greatest at the time the dough is placed in the oven. From the initial gassing curve a single gassing figure is obtained by the following equation:

$$\frac{T \times H}{1000}$$

T is the time taken to reach the highest peak in minutes and H the height of the peak in milliliters of pressure before the end point, or a definite falling off in pressure, is observed. The gassing figure arrived at in this manner agrees very closely with the diastatic figure of Kent-Jones (1936).

This figure is then applied to a scale to show the amount of sugar or malt solution required, if any, to insure an adequate gassing rate. In this regard it has been shown that an increase of sugar beyond the fermentation requirements is necessary to insure a pleasing crust color in the baked loaves. The sugar or malt solution is made up to a definite strength, according to requirements. The solution being used at the present time in the Cereal Division Laboratory is made up so that 10 ml contains 4% sucrose and 0.3% diastatic malt (250°L). The amounts added to doughs will vary from 0 to 10 ml in increments of 0.2 ml. Low-gassing flours may require the full 10 ml, while high gassers may not require any. This scale has been found to take care of the gassing variabilities found in all the Canadian wheats tested to date.

Like most biological tests, duplication of results is not always arrived at with mathematical precision, but results of a statistical analysis on a series of gas figures from a number of flours run on the machine show the error range to be below 10%, which is accurate enough for routine work. The apparatus, although only a rough working model, has been used for routine test work for the past two years with excellent success. Yeast, as shown by Eisenberg (1940), is an important variable and changes due to yeast supply can be checked by running an initial curve on a known standard flour each time a fresh supply of yeast is received.

Summary

An apparatus for recording automatically the rate of gas produced in fermenting bread doughs is described. The data taken from curves made by this machine have been successfully applied to test baking. On the basis of these data, routine sugar-requirement corrections have been made on flours baked by the modified A.A.C.C. malt-bromate-phosphate baking method in the laboratories of the Cereal Division for the past two years.

Curves have been presented showing the influence of salt and yeast, which demonstrate its value in measuring the effect of various ingredients on gassing properties of fermenting flour doughs.

Acknowledgment

Acknowledgment is made to numerous members of the Central Farm Staff for their assistance in the construction of the working model, also to W. D. McLennan for machining fine mechanical parts and to George Ensell, Glass Technologist of the Department of Mines and Resources, who fabricated all the glass detail.

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THE EFFECT OF VARIETY AND ENVIRONMENT ON THE EQUILIBRIUM MOISTURE CONTENT OF SOYBEAN SEED [1]

A. C. BECKEL and J. L. CARTTER [2]

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The study of the effect of variety and environment on the equilibrium moisture content of soybean seed is of interest because of its direct relationship to the problems of storage and preservation. These general problems have been indicated by Humphries and Hurst (1935). Differences would normally be expected, in view of the fact that varieties of soybeans have been found to vary in all of the major chemical constituents studied thus far. It seemed probable that such a study of equilibrium moisture content would also determine whether the routine moisture determination could be eliminated from the analytical program without introducing an appreciable error in calculating results to a uniform moisture basis. Such an elimination would result in a considerable saving in time and expense.

The samples represented eight varieties, each of which was grown at five locations in the North Central Region. The varieties were Peking, Mandarin, Scioto, Manchu, Mukden, Dunfield A, Dunfield B, and Illini. They were grown at Ames, Iowa; Columbia, Missouri; Urbana, Illinois; Lafayette, Indiana; and Columbus, Ohio. A complete description of their origin and agronomic history is included in a recent publication by Cartter and Hopper (1942).

The experimental procedure consisted in placing a 3-g portion of the seed of each sample in a tared metal dish on a table in a constant-humidity, constant-temperature storage room and determining the changes in weight. This constant-humidity storage room had been designed for the close control of both humidity and temperature by Cartter [3] and the performance at all of the humidities imposed was adequate. An analytical balance was placed in the room, and the weighings were made under the conditions imposed at the time. A correction for the change in surface moisture of the dishes was made by following the change in weight of an empty dish.

From the shape of the curve representing the approach of the moisture content to the true equilibrium moisture content, it was apparent that when the weight of a sample was constant for several

[1] Contribution from the U. S. Regional Soybean Industrial Products Laboratory, Urbana, Illinois, a cooperative organization participated in by the Bureaus of Agricultural Chemistry and Engineering and Plant Industry of the Agricultural Research Administration, U. S. Department of Agriculture, and the agricultural experiment stations of the North Central states of Illinois, Indiana, Iowa, Kansas, Michigan, Minnesota, Missouri, Nebraska, North Dakota, Ohio, South Dakota, and Wisconsin.

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[3] To be published.

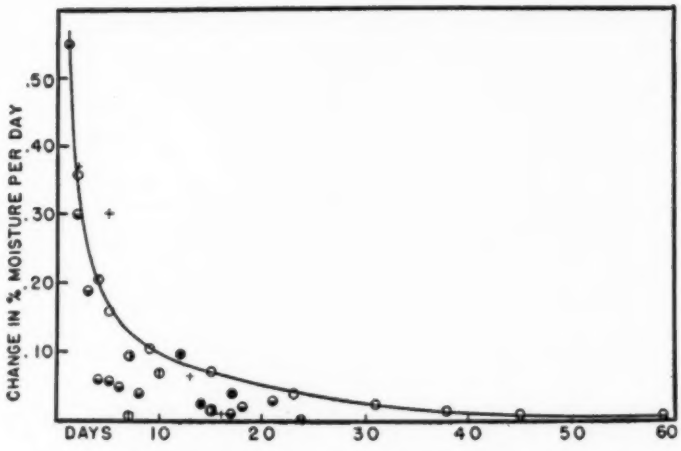

Fig. 1. Rate of approach to equilibrium moisture content of soybean seed

	Change in rh	Change in moisture content
Open circles (0% −18% rh)	↑18%	2.84%
Solid circles (18% −39% rh)	↑11%	1.36%
Plus signs (39% −60% rh)	↑21%	2.23%
Divided circles (60% −43% rh)	↓17%	1.41%
Circles with lower half filled (43% −28% rh)	↓15%	1.24%
Circles with upper half filled (28% −13% rh)	↓15%	0.94%

days, the asymptotic approach to the equilibrium moisture content made a further period of conditioning inadvisable. This behavior has been utilized by Kelley (1940) and Bailey (1920). The small separation of the increasing and decreasing moisture curves is an indication of the failure to reach complete equilibrium, and the true equilibrium content probably lies at some intermediate value. It is probably de-

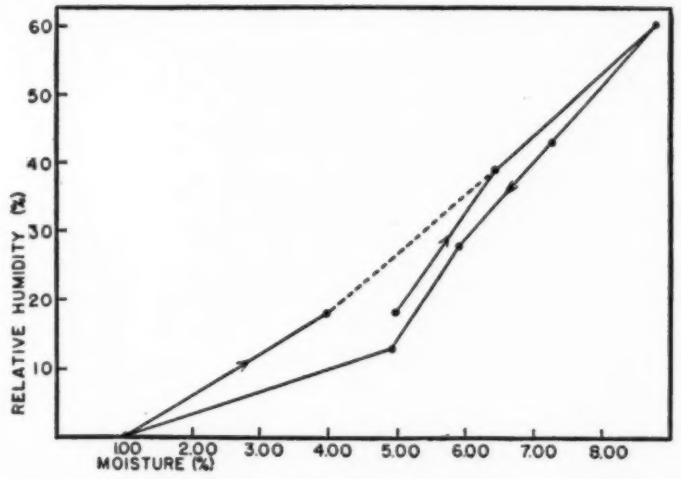

Fig. 2. Absorption and desorption of moisture by soybean seed. Average for all samples studied.

pendent on the sample's previous moisture content, as Auker *et al.* (1942) observed in the case of wheat flour and Urquhart (1929) observed in the case of cotton.

The humidity levels imposed on the samples at 70°F were, in the order of their application, 18%, 39%, 43%, 60%, 28%, 13%, 0%, and 18% relative humidity. The so-called 0% level was obtained by placing the samples in desiccators over phosphorous pentoxide, and, while the humidity prevailing was extremely low, the value obtained actually represented the equilibrium established between the samples and the desiccant. Subsequent to the final 18% level, the samples were

TABLE I

EQUILIBRIUM MOISTURE CONTENTS OF THE SEED OF EIGHT VARIETIES OF SOYBEANS GROWN AT FIVE LOCATIONS DURING 1940, TOGETHER WITH THE STANDARD DEVIATIONS OF THE DETERMINATION AT EACH STORAGE ROOM HUMIDITY, AT 70°F TEMPERATURE

	18%	39%	60%	43%	28%	13%	0%	18%	Mean
COLUMBUS, OHIO									
Dunfield A	4.70	5.98	8.22	6.95	5.61	4.67	0.97	3.75	5.11
Manchu	4.89	6.30	8.61	7.13	5.72	4.71	1.08	3.86	5.29
Illini	5.12	6.56	8.92	7.33	5.96	4.98	0.68	4.16	5.46
Dunfield B	4.75	6.20	8.64	7.01	5.57	4.61	1.01	3.76	5.19
Mukden	4.54	5.83	8.04	6.88	5.51	4.53	1.13	3.42	4.98
Scioto	4.93	6.30	8.92	7.38	6.00	5.09	1.16	4.22	5.50
Mandarin	4.66	5.97	7.92	6.90	5.57	4.57	1.13	3.55	5.03
Peking	5.42	6.96	9.32	7.78	6.28	5.20	1.12	4.21	5.79
Mean	4.88	6.26	8.57	7.17	5.77	4.79	1.03	3.87	5.29
AMES, IOWA									
Dunfield A	4.64	6.02	8.36	6.90	5.48	4.52	0.88	3.72	5.06
Manchu	4.84	6.22	8.56	7.11	5.68	4.70	0.99	3.82	5.24
Illini	4.80	6.23	8.81	7.09	5.65	4.73	0.80	3.98	5.26
Dunfield B	4.81	6.20	8.55	7.07	5.64	4.69	0.96	3.84	5.22
Mukden	4.74	6.09	8.18	7.01	5.61	4.63	0.93	3.59	5.10
Scioto	4.71	6.06	8.57	7.03	5.65	4.77	0.94	3.90	5.20
Mandarin	5.16	6.61	8.87	7.47	6.04	5.01	0.84	4.07	5.51
Peking	5.32	6.80	9.08	7.71	6.23	5.14	1.07	4.13	5.68
Mean	4.88	6.28	8.62	7.17	5.75	4.77	0.93	3.88	5.28
LAFAYETTE, INDIANA									
Dunfield A	4.90	6.37	8.82	7.22	5.74	4.76	1.18	3.89	5.36
Manchu	4.80	6.31	8.73	7.08	5.63	4.65	0.84	3.76	5.22
Illini	4.72	6.24	8.72	6.95	5.53	4.58	0.77	3.85	5.17
Dunfield B	4.59	6.06	8.45	6.88	5.44	4.48	1.09	3.63	5.08
Mukden	4.86	6.32	8.47	7.13	5.70	4.70	1.16	3.65	5.25
Scioto	5.09	6.50	9.06	7.39	5.95	4.94	0.91	4.06	5.50
Mandarin	5.14	6.62	8.77	7.42	6.01	4.99	0.99	4.04	5.50
Peking	5.41	6.98	9.19	7.81	6.31	5.23	1.14	4.20	5.78
Mean	4.94	6.49	8.78	7.23	5.79	4.79	1.01	3.88	5.36

TABLE I—*Continued*

	18%	39%	60%	43%	28%	13%	0%	18%	Mean
COLUMBIA, MISSOURI									
Dunfield A	4.88	6.33	8.52	7.18	5.75	4.75	1.06	3.82	5.29
Manchu	4.62	6.08	8.27	6.86	5.48	4.52	0.95	3.66	5.05
Illini	4.78	6.28	8.78	7.11	5.66	4.70	0.81	3.89	5.25
Dunfield B	5.32	6.86	9.16	7.68	6.23	5.18	0.99	4.21	5.70
Mukden	5.36	6.94	9.34	7.88	6.42	5.40	1.33	4.28	5.87
Scioto	4.97	6.42	8.73	7.22	5.79	4.81	0.97	3.92	5.35
Mandarin	5.51	7.21	9.63	8.12	6.56	5.43	1.04	4.48	6.00
Peking	5.08	6.62	8.82	7.42	6.00	4.97	1.04	3.98	5.49
Mean	5.06	6.59	8.91	7.43	5.99	4.97	1.02	4.03	5.50
URBANA, ILLINOIS									
Dunfield A	4.82	6.35	8.80	7.05	5.80	5.03	1.04	4.08	5.37
Manchu	5.21	6.64	9.01	7.43	6.07	5.28	1.08	4.24	5.62
Illini	4.82	6.44	8.90	7.21	5.95	5.03	0.88	4.08	5.41
Dunfield B	5.05	6.47	8.60	7.18	5.97	5.12	0.99	4.12	5.44
Mukden	5.19	6.51	8.86	7.62	6.23	5.33	1.28	4.26	5.66
Scioto	5.14	6.61	9.13	7.52	6.26	5.07	1.12	4.20	5.63
Mandarin	5.54	6.91	8.64	7.77	6.33	5.68	1.58	4.53	5.87
Peking	5.13	6.62	9.63	7.53	6.20	5.29	1.05	4.17	5.70
Mean	5.11	6.57	8.95	7.41	6.10	5.23	1.13	4.20	5.59
AVERAGE AT 5 LOCATIONS									
Dunfield A	4.79	6.21	8.54	7.06	5.68	4.75	1.03	3.85	—
Manchu	4.87	6.31	8.64	7.12	5.72	4.77	0.99	3.87	—
Illini	4.85	6.35	8.83	7.14	5.75	4.80	0.79	3.99	—
Dunfield B	4.90	6.36	8.68	7.16	5.77	4.82	1.01	3.91	—
Mukden	4.94	6.34	8.58	7.30	5.89	4.92	1.17	3.84	—
Scioto	4.97	6.40	8.88	7.31	5.93	4.94	1.02	4.06	—
Mandarin	4.87	6.31	8.64	7.12	5.72	4.77	0.99	3.87	—
Peking	5.28	6.80	9.21	7.65	6.20	5.17	1.08	4.14	—
AVERAGE FOR ALL VARIETIES AT ALL LOCATIONS									
	4.97	6.43	8.77	7.29	5.88	4.91	1.02	3.97	—
STANDARD DEVIATIONS FOR EACH HUMIDITY LEVEL									
Standard deviation	0.27	0.32	0.38	0.32	0.30	0.30	0.16	0.25	—
Maximum deviation from the mean	0.57	0.68	0.86	0.83	0.68	0.77	0.46	0.56	—
Coef. of variability	5.43	4.98	4.33	4.39	5.10	6.11	15.68	6.30	—

brought to the laboratory and the oven moisture value was determined by heating to 130°C for two hours. It can be seen (Table I) that in terms of the oven-moisture value the samples still retained about 1% of moisture after the long exposure to the atmosphere over phosphorous pentoxide. This value may be related to the bound water of the seed.

It has recently been shown by Bernhardt (1941) that crystalline egg albumin retains about 2% of water when stored over phosphorous pentoxide and that this water is driven off only by heating above 100°C.

Analysis of variance (Fisher, 1933) of the data (Table II) shows

TABLE II

ANALYSIS OF VARIANCE OF DATA ON EQUILIBRIUM MOISTURE CONTENT OF SOYBEAN SEED AS AFFECTED BY HUMIDITY OF THE STORAGE ROOM

Source	Degrees of freedom		Mean square	
Total	319		—	
Varieties	7		0.98	
Locations	4		1.15	
Humidities	7		215.78	
Varieties × locations	28	301	0.37	0.052
Varieties × humidities	49		0.04	
Locations × humidities	28		0.03	
Varieties × locations × humidities	196		0.014	

significant variance in moisture content among the varieties studied. Variance due to locations was greater than that due to varieties, though not by a statistically significant amount. Variance due to humidities was very large, as would be expected among the wide humidity values chosen for study.

The varieties × locations interaction was large with respect to the second order of interaction, varieties × locations × humidities, indicating that the varieties reacted differently to the five locations or environmental conditions during the one season. Also the varieties × humidities and locations × humidities interactions were highly significant, though of much smaller magnitude than the varieties × locations interaction. This measure of the influence of the factors of variety, climate, and of the subsequent storage conditions on the equilibrium moisture content of soybean seed is of value in predicting whether the routine moisture determination may be eliminated from the analytical program, when data on composition are calculated to a uniform moisture basis. The individual moisture contents of the samples at the indicated storage room humidities are given in Table I. These samples were comprised of varieties differing in their normal dates of maturity as well as in the percentage composition of protein and oil, grown at five widely separated locations in the soybean belt.

The standard deviations and coefficients of variability of the samples at each humidity level include all varietal and location variance. This table (Table I) shows a standard deviation of only 0.27% moisture for all samples originally stored at 18% relative humidity and 0.25% for the same samples when returned to that level from a low humidity. In each case, at this humidity level, the maximum deviation from the

mean for any sample was not more than 0.57% moisture. On the basis of these deviations, Table II shows that the error introduced by using the mean value for the moisture content, when calculating analyses to the dry basis, would be less than half of the usual allowable experimental error in the case of the maximum deviation and less than one-third of the allowable error in the case of the standard deviation.

TABLE III

ERROR INVOLVED IN CALCULATION OF ANALYSES TO THE DRY BASIS

Determination	Approximate percent	Error	
		At maximum deviation	At standard deviation
	%	%	%
Potassium	1.75	0.005	0.003
Phosphorous	0.600	0.005	0.003
Calcium	0.300	0.005	0.003
Lipids	20.00	0.10	0.06
Nitrogen	6.00	0.03	0.018
Mean moisture at 18% relative humidity = 4.97%			

A similar calculation would show all of the relative humidity levels, except 60%, to be satisfactory.

Obviously, the moisture determination may be eliminated for samples that have a similar agronomic and early storage history and have been stored under uniform humidity and temperature conditions for a period of about a month, provided there is no significant and irregular variation in the moisture content during the grinding and mixing of the sample. Samples ground in the constant-humidity room and in the atmosphere of the laboratory showed a uniform variation in moisture of less than 0.10%, indicating that a standardized grinding procedure would not introduce a significant and irregular variation in moisture content. It has been found in this laboratory that a relative humidity of about 18% at 70°F reduces the water content of soybeans below the 5.5% level where, as has been shown by the Soybean Analysis Committee of the American Oil Chemists Society (1939), a more easily reproducible oil determination is possible when using the official A.O.C.S. method. Higher moisture contents cause the extraction of other materials in addition to the triglycerides.

Summary

Approximate equilibrium moisture content of the seed of eight strains of soybeans grown at five locations in the North Central Region was determined at eight relative humidity levels at 70°F. Significant variance was observed due to variety and location. Including all sample variance at each of the humidity levels, the standard deviation

ranged from 0.163 at the low humidity level to 0.382 at 60% relative humidity.

From data at 18% relative humidity and 70°F, it has been shown to be possible to eliminate the routine moisture determination without affecting the accuracy of conversion of other analytical data to the dry basis.

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DIRECT DETERMINATION OF FERMENTATION RATES IN DOUGH

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(Read at the Annual Meeting, May 1942)

In dough fermentation certain quantities of sugar, nitrogen compounds, and accessory factors are used up in producing a corresponding total amount of gas or expansion. This total, when used in reference to the capacity of the flour to produce it, is commonly designated as gassing power. On the other hand, the *rate* of gas production, or the rate of fermentation or expansion, is the derivative of the former function, and, as is now well recognized, varies greatly as the fermentation proceeds.

A number of investigators have studied average rates, *i.e.*, have determined $\Delta V/\Delta t$ from integrated rate-of-gas-production measure-

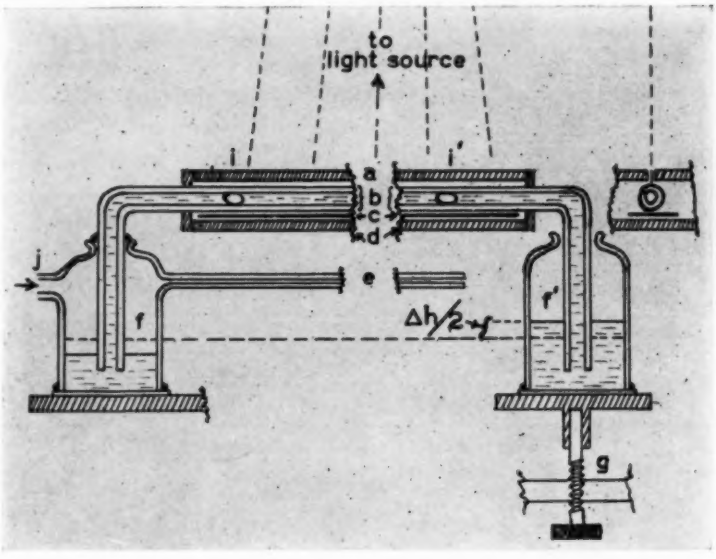

Fig. 1. Fermentation rate meter. The apparatus comprises essentially a sensitive differential manometer with a kymograph drawing standard Ad-type developing out (Azo) photographic printing paper, *c*, beneath the bubble tube, *b*. The shields *a* and *d* are light-tight, carrying slits parallel to the tube through which light reaches the paper. Gas from the fermentation jar enters the cup *f* at *j*, and flows out to the atmosphere through capillary tube *e*. The capillary resistance creates a difference in pressure, Δh, and manometric fluid (kerosene of sp g 0.810) flows from *f* to *f'* until the difference in levels equals Δh. The bubble in the tube *b* assumes a new position *i'*, corresponding to Δh. From Poiseulle's law we have that

$$\Delta h = \frac{k\eta l}{\pi g d^4} = v,$$

where k is a constant, η is the viscosity of the gas, l the length of the capillary, *e*, and d is its diameter. The rate of flow of gas through the capillary is $v = dV/dt$. There is thus a direct relationship between the position of the bubble and the instantaneous rate of gas production. This relationship is linear if the cross sectional areas of the two cups *f* and *f'* are equal and the diameter of tube *b* is uniform. These conditions may be met if precision-bore tubing is used. The complete equation for the instrument then becomes

$$\Delta i = \frac{K\eta l v}{2d^4\rho}\left(\frac{R^2 - r_0^2}{r_i^2}\right),$$

where if Δi is the distance of bubble travel in cm,
d the diameter of capillary tube *e* in mm,
ρ the density of manometric fluid in g/ml,
l the length of capillary tube *e* in cm,
η the viscosity of gas in c.g.s units (for air $\eta = 172 \cdot 10^{-6}$, for CO_2 $\eta = 160 \cdot 10^{-6}$),
R the radius of cups *f* and *f'* in any units,
r_i and r_0 the inside and outside diameters of bubble tube *b* in the same units,
then $K = 6.88 \pm 2\%$.

Since the viscosities of air and CO_2 differ by about 7.5%, an error is introduced when gas escapes from the dough, but this may be minimized by calibrating with a mixture of the gases. Precision-bore tubing may be obtained with a tolerance of 0.01 mm, and if a capillary of say 0.9 mm is used this will introduce an error of about 5.7%. Under certain conditions a tolerance of 0.001 mm has been achieved; the corresponding error is 1.3%. Other sources of error in design are small compared to these two if precision-bore tubing is used. The values of these quantities for the instrument used in this work are approximately as follows:

Δi_{max} = 25.4 cm
ρ = 0.810 g/ml (kerosene with red dye)
d = 0.865 mm
l = 20 cm
R = 15.98 mm
r_0 = 5.8 mm
r_i = 2.52 mm

For these dimensions Δh for full capacity is about 1 cm of kerosene of density 0.810, or about 0.6 mm Hg. Under steady-state conditions this may be responsible for an error of about 0.08%, which is negligible. When the rate is rapidly changing, however, there is an instrument lag which is proportional to the volume of the fermentation chamber and the acceleration, *i.e.* to $V_0/P_0(d^2V/dt^2)$. It is thus advisable to keep the volume of the fermentation jar small in relation to Δh.

The units of the apparatus may conveniently be sealed together with DeKohtinsky cement, which s inert to kerosene. Screw *g* is used for adjusting the zero position of the bubble.

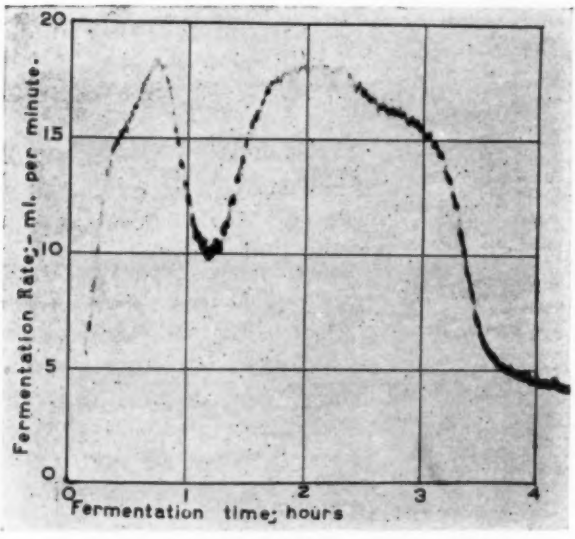

Fig. 2. Sponge-type fermentation for a high-protein flour.

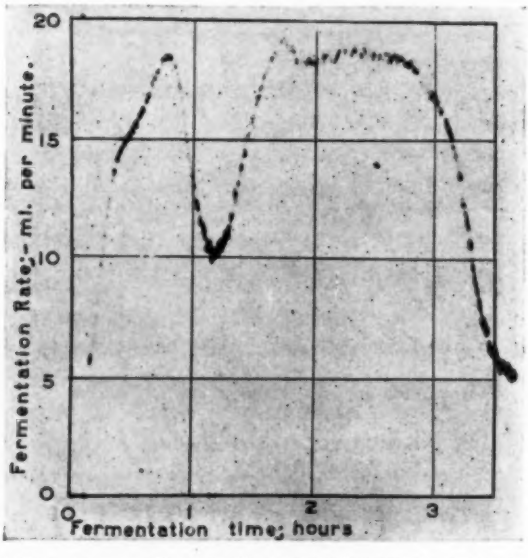

Fig. 3. Same as Figure 2 with 0.05% $NH_4H_2PO_4$ in the batch.

ments as a function of time, where Δt has been as small as 5 minutes (cf. Eisenberg, 1940). The measurement of instantaneous rates and accelerations, however, presents certain difficulties. James and Huber (1928) allowed the water displaced from a tank by the gas produced during fermentation to flow through an orifice under the gravitational

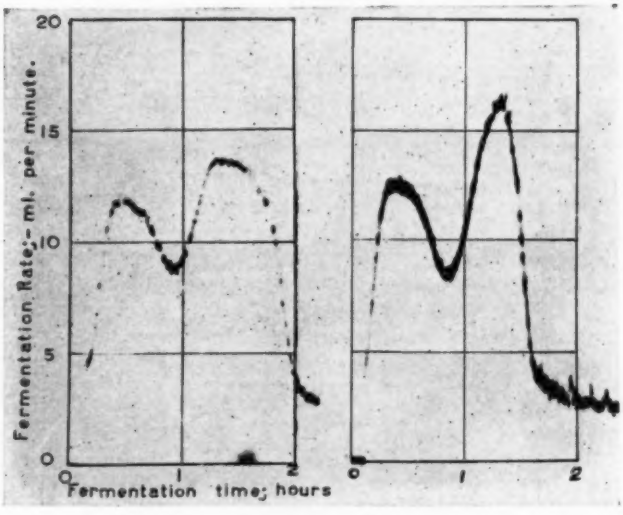

Fig. 4. Sponge-type fermentation for a low-protein flour; without and with 0.05% $NH_4H_2PO_4$ respectively.

field. A one-to-one correspondence existed between the instantaneous rate of gas production and the height of water above the orifice, which was thus taken as a measure of the former. A recording device was used, but mechanical difficulties were troublesome. However, readings taken at intervals and plotted against time showed characteristic waves and peaks in some cases, which confirmed results obtained at these Laboratories on rate changes taking place during transference of the fermentation from one sugar to another. These peaks were later identified with specific dough conditions by Larmour and Bergsteinson (1936).

In order to achieve sufficient sensitivity the principle of the differential manometer was adapted to this problem, and an optical recording system was developed. The apparatus is shown diagrammatically in Figure 1. Any desired degree of sensitivity can be obtained, and individual bubble bursts from the surface of the dough recorded. In effect, this represents the "noise level" of the dough: equalization of random inequalities of pressure just beneath the surface. The apparatus is particularly well suited for studying the fermentation intensity at any moment during dough time.

Typical Results

Typical sponge-type fermentations at a constant temperature of 30°C are shown in Figures 2, 3, and 4. The ordinate represents the rate for 6 g of yeast in all cases. Figures 2 and 3 without and with,

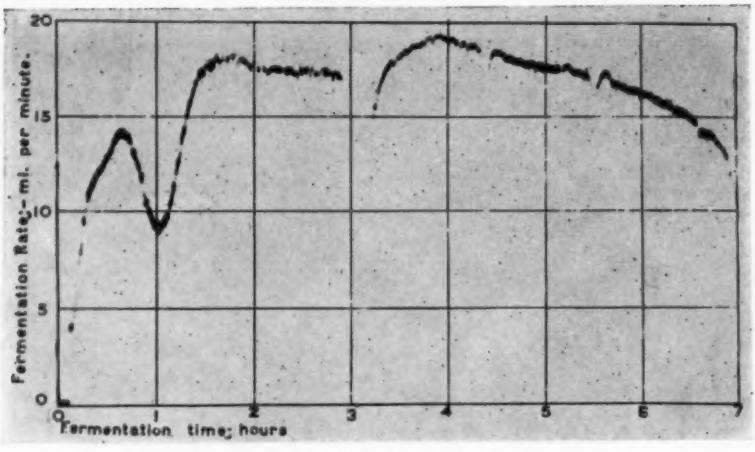

Fig. 5. Laboratory sponge and early dough. Constant temperature.

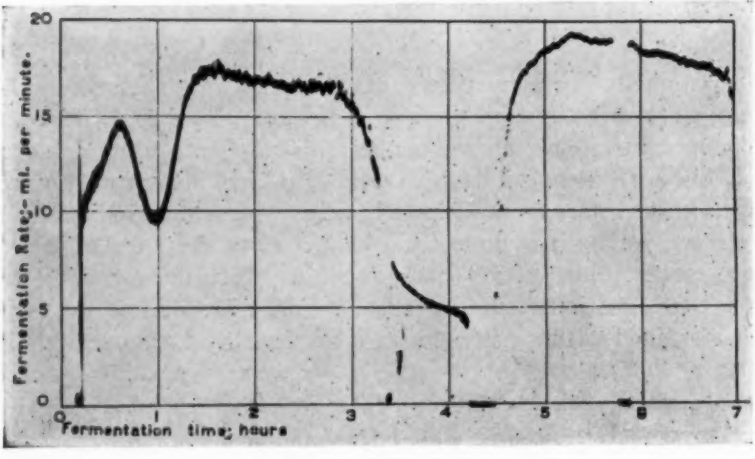

Fig. 6. Laboratory sponge and medium dough.

respectively, 0.05% $NH_4H_2PO_4$ are characteristic of high-protein flours (15% protein) with an adequate amount of nutrient substances. The ammonium phosphate exerts only a slight stimulating effect in the later stages of fermentation.

The transference from sucrose (or hexose) fermentation to maltose fermentation as shown by Larmour and Bergsteinson (1936) is quite well marked, as is the rapid decline to the diastatic level after the supply of preformed and diastatically liberated sugars is exhausted. Figure 4 shows the same type of fermentation for a low-protein flour (9%). Here the addition of 0.05% of $NH_4H_2PO_4$ produces a striking accelera-

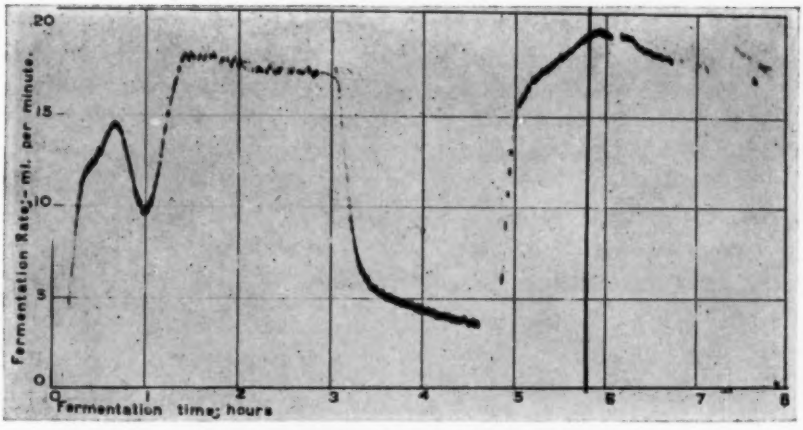

Fig. 7. Laboratory sponge and late dough.

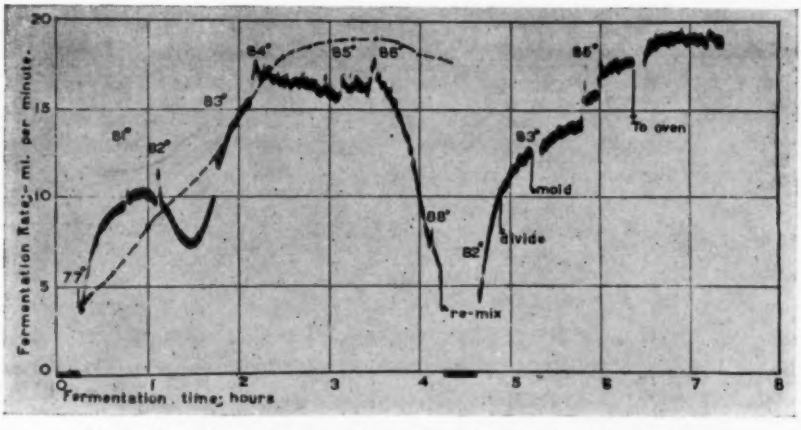

Fig. 8. A medium-cool commercial sponge and dough. The dotted line gives the height of the sponge in arbitrary units.

tion in the later stages, due largely to the flour's inherent deficiency in available amino derivatives. The low gassing power, as indicated by the area under the curve, is quite noticeable.

Fermentation rate during a complete laboratory sponge and dough procedure (2% yeast based on total flour) at constant temperature (30°) is shown in Figures 5, 6, and 7. One reason for the popularity of this method in commercial practice is here indicated. The dough fermentation rates are practically identical, although sponge time is varied from just before the "break" in fermentation rate (sugar exhaustion) or "drop" of the sponge, to 1½ hours after this point. Addition of new food substances in the fresh topping flour permits the fermentation to proceed at its maximum rate for a considerable time after remixing.

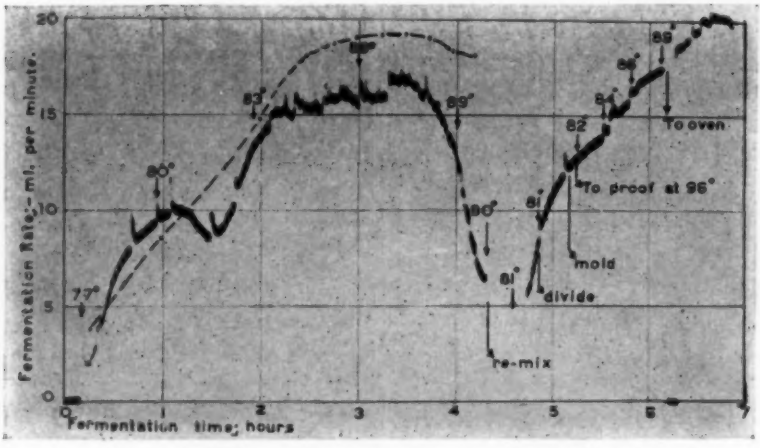

Fig. 9. A warm commercial sponge and dough.

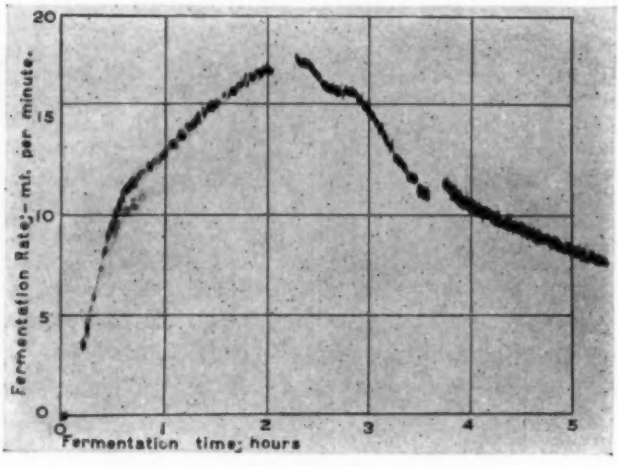

Fig. 10. Straight-dough type of fermentation.

But in the commercial dough the baker permits the temperature to vary widely during the process, and controls it by cooling during the re-mix—then raises it rapidly by proofing in a high-temperature proof box. Figures 8 and 9 show two such commercial runs, and the changed fermentation characteristic is quite striking. A rapidly rising rate during the dough stage is induced by the high temperature of the proof box (90°F), although the dough is still five or six degrees below this temperature when proof is completed.

A characteristic straight-dough fermentation is shown in Figure 10. The addition of 5% of sucrose to the mix eliminates the inflection

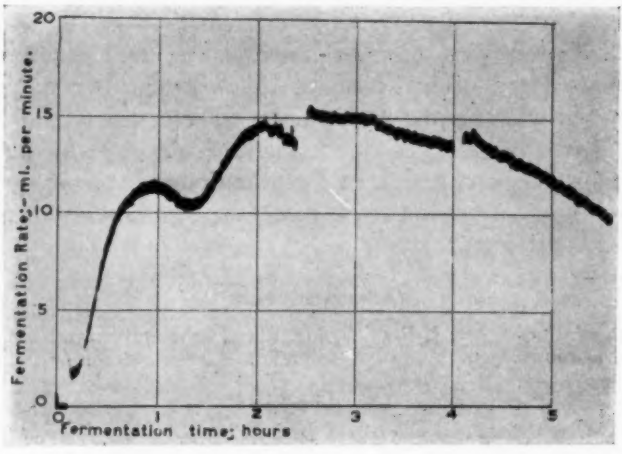

Fig. 11. Straight-dough type of fermentation with all malt sirup as sugar supplement.

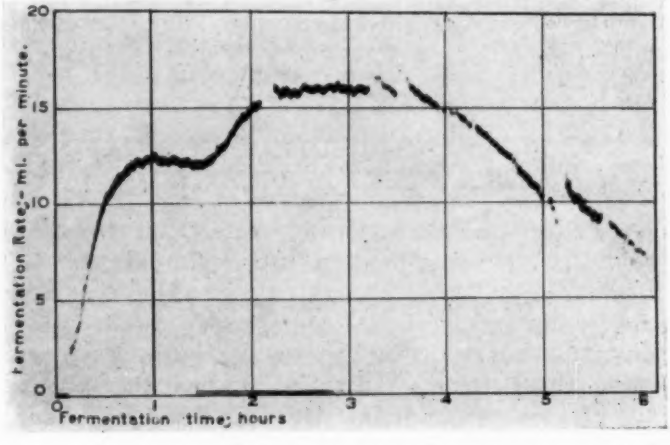

Fig. 12. Straight-dough type of fermentation with pure maltose as sugar supplement.

noted in the sponge-type fermentation, and after a single maximum is reached the rate gradually declines, the deceleration depending on the accessory yeast food factors in the dough at that time. The current interest in sugar substitutes makes it pertinent to inquire concerning the effect of other sugars. Figure 11 shows the effect of malt syrup and Figure 12 that of pure maltose. The effect is not pronounced in the sponge and dough process, but those bakers utilizing the straight-dough method may observe some differences.

Summary

A sensitive recording differential manometer used to measure rates of gas flow is described and fermentation rate curves obtained under various conditions are presented. The transference of fermentation from sucrose to maltose in sponge fermentation is illustrated. A rising fermentation rate is characteristic of the dough stage in commercial sponge and dough procedures, which differs from laboratory fermentations at constant temperature.

Acknowledgment

The cooperation of Mr. J. Freilich, Mr. Floyd Schoonover, and Mr. H. Kothe is gratefully acknowledged.

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SELENIUM DISTRIBUTION IN MILLED SELENIFEROUS WHEATS [1]

A. L. Moxon,[2] O. E. Olson,[2] E. I. Whitehead,[2] R. J. Hilmoe,[2] and Stewart N. White [3]

(Received for publication November 23, 1942)

The first published report that wheat contained selenium was made by Robinson (1933) after he had analyzed wheat that Franke (1934) had found to be toxic to animals. Although this first report involved wheat that had been grown in South Dakota, subsequent reports have shown that seleniferous vegetation occurs in many parts of the world. Robinson (1936) has reported the selenium content of wheat grown in South America and other parts of the world. Byers and Lakin (1939), Thorvaldson and Johnson (1940), and Williams, Lakin, and Byers (1941) have reported on the selenium content of wheat from Canada. Lakin and Byers (1941 and 1941a) and Williams, Lakin, and Byers (1941) have reported seleniferous vegetation in most of the states west of the Mississippi river.

[1] Approved for publication by the Director of the South Dakota Agricultural Experiment Station, as contribution No. 169 of the Journal Series.

[2] South Dakota Agricultural Experiment Station, Brookings, South Dakota.
[3] Tri-State Milling Company, Rapid City, South Dakota.

Although there are a few localized areas in South Dakota that produce highly seleniferous vegetation, most of the seleniferous areas in this state produce wheat of about the same selenium content as that grown in seleniferous areas of other Great Plains and western states.

Even though the occurrence of selenium in wheat is rather widespread, it appears that the selenium analyses of the milled products of only one experimentally milled sample of seleniferous wheat have been published. This sample was milled in 1931 [4] for bioassay studies and the selenium analyses were made at a later date and published by Painter and Franke (1940). Horn, Nelson, and Jones (1936) studied the distribution of the toxic factor in the various fractions of milled seleniferous wheat but did not report the selenium content of the fractions. They reported that the toxic factor was quite evenly distributed in the various fractions, whereas the analysis of the milled sample (Painter and Franke, 1940) indicated that the selenium content of the bran and the middlings was much higher than that of the flour. Since the method described by Klein (1941) for determining selenium is much more accurate than methods used heretofore for plant materials, it seemed desirable to investigate the distribution of selenium in a few experimentally milled seleniferous wheats.

Experimental

Four samples of dark northern spring wheat grown in seleniferous areas of the state in 1941 were milled. The samples were prepared for milling by scalping and dry scouring. The clean dry wheat was made up to 16% moisture with water at 40°C and tempered for 18 hours.

The milling was carried out on a Buhler experimental mill,[5] which is a three-break and three-reduction mill. The first break, second break, and third break are clothed with 28 wire, 36 wire, and 40 wire, respectively, and in addition each has a 10XX silk. The first and second reductions are clothed with a 44 wire and a 54 wire scalp and each have two 10XX silks. The third reduction has a 10XX silk. The degree of grinding was set for good milling practice, yield, and clean-up. The milling-room temperature was 80°F with a relative humidity of 55%.

The fractions were all analyzed for moisture and selenium, and the fractions from sample No. IV were also analyzed for nitrogen (Kjeldahl) and sulfur.

Selenium was determined by the method described by Klein (1941) with a few minor modifications. Sulfur was determined by the Parr Bomb method (1939), and Kjeldahl nitrogen determinations were made with copper sulfate as the catalyst.

[4] Milled by Dr. C. H. Bailey, Division of Agricultural Biochemistry, University of Minnesota.
[5] Located in the laboratories of the Tri-State Milling Co., Rapid City, S. D.

TABLE I

THE PERCENTAGE OF WHOLE SAMPLE IN EACH MILL FRACTION
(Moisture-free basis)

Fractions	Wheat number			
	I	II	III	IV
	%	%	%	%
First-break flour	6.9	8.8	4.0	6.4
Second-break flour	3.8	3.4	2.5	3.1
Third-break flour	1.8	1.6	1.3	1.5
First-reduction flour	37.2	36.5	23.7	33.8
Second-reduction flour	14.1	14.0	18.6	17.6
Third-reduction flour	2.8	2.8	9.5	4.9
Bran	26.2	24.9	26.8	24.3
Shorts	7.2	8.0	13.7	8.3

TABLE II

SELENIUM CONCENTRATION IN MILL FRACTIONS
(Parts per million, moisture-free basis)

Fractions	Wheat number			
	I	II	III	IV
	ppm	*ppm*	*ppm*	*ppm*
First-break flour	4.1	5.4	51.4	20.1
Second-break flour	4.7	5.8	56.7	21.1
Third-break flour	4.8	5.7	62.5	20.3
First-reduction flour	3.8	3.4	52.9	19.0
Second-reduction flour	4.6	4.3	52.9	19.0
Third-reduction flour	4.4	3.8	55.4	17.2
Bran	5.9	8.7	88.4	33.4
Shorts	5.5	6.3	77.2	24.8
Wheat [1]	4.8	5.8	63.0	23.3

[1] Fractions recombined on percentage basis (Table I), carefully mixed and analyzed.

TABLE III

PERCENTAGE OF TOTAL SELENIUM IN EACH MILL FRACTION
(Moisture-free basis)

Fractions	Wheat number			
	I	II	III	IV
	%	%	%	%
First-break flour	5.98	8.90	3.01	5.60
Second-break flour	3.85	3.71	2.18	2.82
Third-break flour	1.92	1.67	1.17	1.30
First-reduction flour	30.13	23.01	19.01	27.86
Second-reduction flour	13.89	11.13	14.72	14.50
Third-reduction flour	2.56	2.04	7.98	3.65
Bran	33.12	40.26	35.87	35.24
Shorts	8.55	9.28	16.06	9.03

Results and Discussion

Table I shows the percentage yields of the various milled fractions. The percentage of bran from each sample was fairly constant, but there was considerable variation in the other fractions, undoubtedly due to the differences in the milling qualities of the wheats.

Table II shows the concentration of selenium in each fraction for the four different samples. There was a great difference in the selenium concentration in the different fractions. The bran was highest in selenium content in all cases and was likewise highest in nitrogen (Table IV), which probably explains the reason for the high selenium content, because it has been shown that the selenium is closely associated with the proteins of wheat. Table III shows the percentage of the total selenium occurring in each milled fraction.

The average selenium content of all the flour fractions, the selenium content of the patent flour, and the percentage of patent flour are shown in Table IV. Table V shows the nitrogen-selenium, sulfur-

TABLE IV

SELENIUM IN FLOUR

(Parts per million, moisture-free basis)

Fractions	Wheat number			
	I	II	III	IV
All flour fractions	4.1	4.05	53.58	19.06
Patent flour [1]	4.08	4.02	53.26	19.09
Percent patent	95.9	96.3	92.6	92.9

[1] Patent flour = first and second break fractions, first and second reduction fractions, and two-thirds of the third reduction fraction.

TABLE V

NITROGEN, SULFUR, AND SELENIUM IN VARIOUS FRACTIONS OF MILLED WHEAT (SAMPLE IV)

Fractions	Percent of total	Nitrogen (moisture-free)	Sulfur (moisture-free)	Selenium (moisture-free)	N/Se[1]	S/Se[1]	N/S[1]
	%	%	%	*ppm*	*moles*	*moles*	*moles*
First-break flour	6.4	2.50	0.190	20.1	6,999	233	30
Second-break flour	3.1	2.84	0.209	21.1	7,643	246	31
Third-break flour	1.5	3.14	0.230	20.3	8,936	290	31
First reduction flour	33.8	2.43	0.177	19.0	7,212	229	31
Second-reduction flour	17.6	2.39	0.186	19.0	7,109	241	29
Third-reduction flour	4.9	2.56	0.187	17.2	8,393	270	31
Bran	24.3	3.36	0.270	33.4	5,667	199	28
Shorts and red dog	8.4	2.93	0.236	24.8	6,670	234	28
Total	100.0	—	—	—	—	—	—
Whole grain	100.0	2.76	0.203	23.3	6,681	215	31

[1] Molecular weights used: sulfur, 32.06; selenium, 78.96; nitrogen, 14.00.

selenium, and nitrogen-sulfur ratios for Sample IV. These results are in close agreement with those published by Painter and Franke (1940).

Samples I, II, and III are below the 1941 state average in protein content, while Sample IV is above the average with 15.73% of protein (N × 5.70). The first three samples contained 13.71%, 13.45%, and 12.80% of protein, respectively.

The average protein content of 13,351 samples of 1941 spring wheat from all parts of the state was 14.23% (N × 5.70).[6]

Summary

Four samples of seleniferous wheat have been milled in an experimental mill and the distribution of selenium in the milled fractions has been determined. Nitrogen-selenium, sulfur-selenium, and nitrogen-sulfur ratios have also been determined for one of the samples.

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FRACTIONATING AND RECONSTITUTING TECHNIQUES AS TOOLS IN WHEAT FLOUR RESEARCH[1]

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Many studies have been made to determine the relative importance of various constituents and fractions of flours differing in bread-making potentialities and properties. Some workers have attempted to correlate flour quality differences with physical and chemical characteristics of their starches. Others have worked entirely with the flour proteins or gluten fractions. An important conclusion is that there is considerable lack of agreement as to which constituent or fraction of the flour is responsible for the differences in quality and properties of the many wheat varieties.

A procedure used in the past has been to separate the gluten and starch by washing with water, after which the two fractions were recombined in various ways to produce a flour or dough which was then baked into bread. The validity of this procedure depends on the assumption that the characteristics of the gluten and starch were not materially altered and that no essential constituents were lost in the wash water. In fractionating and reconstituting studies carried out in this laboratory, the constituents contained in the wash water were recovered and in many instances recombined with the gluten and starch fractions, thus giving a dough containing all the constituents originally present in the flour.

This technique has been used (1) to locate the constituent responsible for flour quality and for the oxidation or bromate requirement of flour, (2) to determine the function and importance of the water-soluble fraction, and (3) to study the relation between loaf volume and protein content in flours of abnormal protein levels, especially from 0% to 8%. The purpose of this paper is to present preliminary data that have been obtained regarding these problems.

Literature Review

Morea (1937) used the wet gluten separated from ordinary baker's sponge to fortify various bakery products, with beneficial results.

Aitken and Geddes (1938) prepared dry glutens by employing a drying temperature of 32°C and a rapid air flow. After reducing the glutens from weak, intermediate, and strong wheats to a flourlike

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fineness, they fortified the original flours with the corresponding glutens in such a manner as to equalize their protein contents. Thus they expected to compare more accurately their relative gluten qualities. The additions of dry gluten resulted in decided improvement in dough-handling properties and loaf appearance, as well as increases in loaf volume and absorption. The crumb texture was either unimpaired or improved but the crumb color was more yellow or grayish-yellow. Later (1939) they prepared seven flours ranging in protein content from 10.5% to 22.7% by enriching the lowest-protein flour with dried gluten. These protein-fortified flours were employed to study the relation between loaf volume and protein content over a wide range without introducing differences in protein character.

Sandstedt, Jolitz, and Blish (1939) recombined wheat starch and gluten to form doughs which were regarded as similar in baking characteristics to the original flour doughs.

Harris (1940) states that the method of drying wet crude gluten as described by Aitken and Geddes (1938) proved entirely feasible, although there was some evidence of alteration in the properties of the dried glutens.

In all of the above studies the water-soluble fraction, necessarily removed in washing the gluten, was discarded.

Material and Methods

Three varieties of wheat, Thatcher, Kharkof, and Chiefkan, known to differ greatly in baking quality characteristics, were used in these studies. Thatcher is a widely grown spring wheat and generally recognized as of excellent quality for bread. Kharkof is typical of the winter wheat grown in the hard winter wheat region and is also known for its good quality characteristics. Chiefkan is generally regarded as being unsatisfactory for bread and in this laboratory has been found to differ materially in quality characteristics from other hard red winter varieties. The Thatcher used in these studies was a composite lot made up of four samples of grain grown in the Northern Great Plains and supplied by the Northwest Crop Improvement Association. The flour milled from it contained 14.9% protein. The Kharkof and Chiefkan were also composite lots of grain grown in comparable experimental trials in the hard red winter wheat region; flours from these wheats contained 15.8% and 14.8% protein, respectively. The various flours were stored at a temperature of 2° to 5°C previous to use in these studies.

The general procedure consisted of mixing the flours for ¼ minute immediately after removal from storage with sufficient distilled water (also at a temperature of 2° to 5°C) to make a dough of the desired

consistency and then permitting the doughs to stand for 15 minutes at 2°C to permit hydration. Each dough was then separated into three fractions: gluten, starch, and water-soluble material. These fractions were then recombined in their original proportions and in various ways, and the doughs baked into bread by the usual methods used in this laboratory.

A further fractionation of the glutens from Kharkof and Chiefkan was made by extracting with ethanol alone, petroleum ether alone, and with several portions of ethanol followed by several portions of petroleum ether. The fatty residues were reclaimed after the solvents were evaporated in a strong current of air at room temperature. Each of the three fatty residues thus obtained from Kharkof gluten was then recombined with the corresponding Chiefkan fat-free gluten, starch, and water-soluble fractions, in their original proportions. Similarly each of the three Chiefkan fatty residues was recombined with its corresponding Kharkof fat-free gluten, starch, and water-soluble fraction. The amount of ground gluten necessary to give a flour having the same protein and total solids as the original (Table I) was extracted with each solvent or combination of solvents. The fatty residue from each extraction was added back to the reconstituted flour after it was dissolved in the 3 g of shortening, which was the amount used in the baking formula for each 100 g of flour. The original flours were also baked into bread for comparison.

The pertinent characteristics of the baking method were: a mixing time considered suitable for each variety, a rich formula containing dried skim milk, and, in most cases, sufficient bromate to meet the requirements of each variety (2 to 4 mg of $KBrO_3$). Additional bakes were also made with different amounts of bromate. In general, single bakes were made because of the difficulties of preparing materials, but the agreement and orderliness of the results indicate their reliability.

In the separation of doughs into various fractions, they were washed with six 75-ml and two 50-ml portions of cold distilled water (2° to 5°C). Each washing was strained through a 40GG flour cloth. The water with its soluble contents was separated from the starch by centrifuging. The liquid fraction, after standing an hour at 2°C to remove foam, was evaporated to a syrupy consistency under reduced pressure at 18° to 20°C. Evaporation was effected by using a water-cooled condenser ($1\frac{1}{2}$-inch diameter) within a large tube ($1\frac{7}{8}$-inch diameter) which was surrounded by an ice bath. The rate of evaporation was 250 ml per hour. The centrifuged moist starch was dried at room temperature with the aid of an electric fan. Frequent subdividing and mixing accelerated drying, which was accomplished in approximately 4 hours. After drying to about 14% moisture the starch was

ground in a Hobart mill to pass through a 10XX flour cloth. When cut into small pieces and spread on waxed paper in front of a fan, the gluten dried in 4 to 6 hours. The dry gluten was ground in a Hobart mill to pass through a 70GG flour cloth; 60% to 75% of it was fine enough to pass through a 10XX flour cloth. All products were stored at 2°C until used.

It is important that the approximate temperature conditions specified be observed if an unaltered water-soluble fraction is to be obtained. The gluten should be ground to fineness approaching that of flour in order to insure normal hydration and consequently normal development of the reconstituted dough during mixing.

Experimental Studies

Three more or less separate studies were carried out over a period of several weeks with the material and methods previously described. The first was designed to locate the factors responsible for flour quality. The second was for the purpose of determining the importance and function of the water-soluble fraction, particularly as related to oxidation or bromate requirement of flour. The third was designed to study the relation between loaf volume and protein content at abnormal protein levels, especially from 0% to 8%. The loaves shown in any one figure were all baked on the same day.

Similarity Between Original and Reconstituted Doughs

For a wheat-flour fractionating technique to be of value, each of the fractions must retain its original characteristics to the extent that when a flour is reconstituted and the usual baking ingredients added,

TABLE I

DATA FOR ORIGINAL AND RECONSTITUTED DOUGHS

Variety	See Figure	Loaf volume	
		Original dough	Reconstituted dough
		cc	*cc*
Kharkof	1	955	945
Kharkof	2	985	990, 960, 990
Kharkof	4	950	955
Chiefkan	1	775	760
Chiefkan	2	815	820, 800, 815
Chiefkan	4	780	760
Thatcher	3	975	970

it will yield a dough and loaf of bread identical (within experimental error) with that obtained from the original flour. The data in Table I are presented to show that such was the case for the fractionating studies reported herein.

The standard deviation for loaf volume between two replicates baked in this laboratory on different days is approximately 15 cc. Therefore, since the volumes in Table I represent single loaves, a difference of approximately 30 cc between the volume of the check and each reconstituted loaf would be required for significance. In no case did the difference reach this magnitude. The average difference was 7 cc in favor of the original flours, which is considerably below the level of significance. The loaves are shown in Figures 1, 2, 3, and 4.

It should be noted that the data in the different figures are not always strictly comparable. The yeast used in the bakes illustrated in Figures 1 and 4 produced a loaf volume of 915 cc with a standard flour, while that used for the loaves illustrated in Figure 2 produced a loaf volume of 950 cc with the same flour. The higher loaf volumes of Figure 2, obtained for Kharkof and Chiefkan, are almost certainly due to the greater strength or dough-developing ability of the yeast used, as shown by Finney and Barmore.[3]

Location of Flour Quality and Exchange of Fatty Material from Gluten Fractions of Varieties Differing in Quality

The two Kharkof and Chiefkan flours previously described were separated into starch, gluten, and water-soluble portions for the purpose of determining the fraction responsible for their quality differences. The analyses of the fractions obtained from Kharkof and the amounts used for reconstitution are given in Table II. The analyses

TABLE II

Analysis of Fractions Obtained from Kharkof Flour and Amounts Used for Reconstitution

Material	Moisture	Protein	Amount used		Protein in amount used
			As is	Dry	
	%	%	g	g	g
Starch	14.3	0.83	76.3	65.3	0.63
Gluten	9.6	72.50	19.9	18.0	14.42
Water-soluble material	90.6	2.24	34.2	3.2	0.75
Reconstituted flour	—	15.80	—	86.5	15.80
Original flour	13.9	15.80	100.5	86.5	15.80

for the other varieties differed only in that the amounts of protein in the starch, gluten, and water-soluble fractions of Chiefkan were less than those in the corresponding fractions of Kharkof, while for Thatcher they were greater. The baking results for the reconstituted doughs in which the Kharkof and Chiefkan fractions were interchanged *one at a time* are shown in Figure 1.

[3] Paper presented at the 28th Annual Meeting of the A. A. C. C., Chicago, 1942. In press.

The results show that the recognized differences in quality between these two varieties were entirely accounted for by differences in their gluten fractions. Thus Kharkof with Chiefkan gluten (Loaf 4) was far poorer than the original Kharkof (Loaf 1) or the fully reconstituted Kharkof (Loaf 2), and equal within the limits of random error to the original Chiefkan (Loaf 8). Similarly, Chiefkan with Kharkof

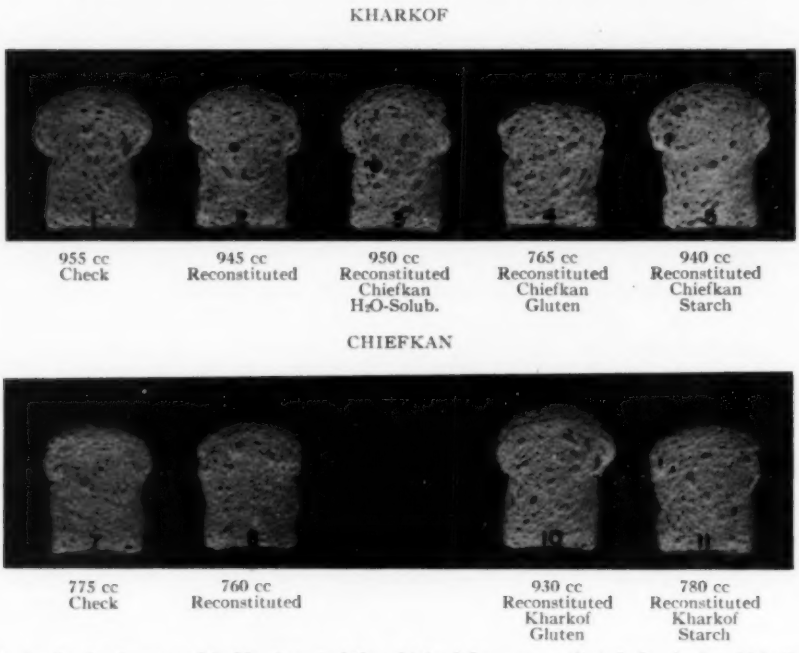


Fig. 1. Loaf volumes and inside characteristics obtained from reconstituted doughs in which the Kharkof and Chiefkan fractions were interchanged one at a time.

gluten (Loaf 10) was far better than either the original or the fully reconstituted Chiefkan (Loaves 7 and 8) and almost equal to the original and fully reconstituted Kharkof (Loaves 1 and 2). The lower volume of Loaf 10, compared to Loaves 1 and 2, is easily accounted for by the fact that the Chiefkan water-soluble and starch fractions each contained about 0.6% of protein, which had a lower loaf-volume-producing ability of 15 to 20 cc than did the same amount of Kharkof protein. (The regression of loaf volume and protein content for Kharkof was about 60 cc for each percent of protein, while that for Chiefkan was about 30 cc.)

Comparisons made with starch and water-soluble fractions interchanged were in accord with those described above. Thus Kharkof with Chiefkan starch (Loaf 5) was substantially equal to the original and to the reconstituted Kharkof, and the Chiefkan with Kharkof

starch (Loaf 11) was substantially the same as the original and fully reconstituted Chiefkan. Also Kharkof with the water-soluble fraction from Chiefkan (Loaf 3) was equal to the original and fully reconstituted Kharkof. Such differences as appeared were clearly within the limits of experimental error, or can be accounted for by lower loaf-volume-producing ability of the protein contained in the Chiefkan starch and water-soluble fractions.

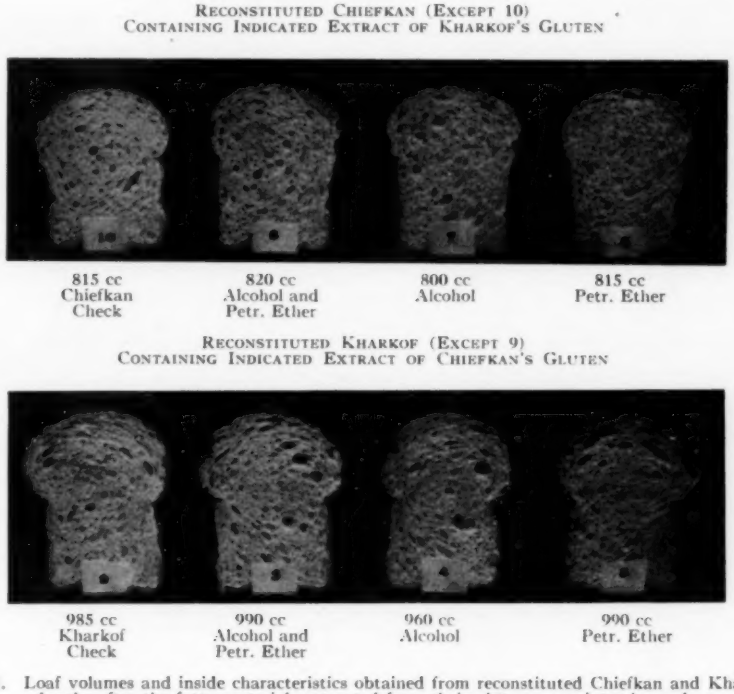


Fig. 2. Loaf volumes and inside characteristics obtained from reconstituted Chiefkan and Kharkof doughs after the fatty materials extracted from their glutens were interchanged.

It has been suggested that baking-quality differences between varieties may be due to the amount or composition of the fatty material. However, if the fatty material of flour has anything to do with the superior bread-making capacity of Kharkof, compared to that of Chiefkan, it must be the fat remaining in the washed gluten, since the data just presented located quality in the gluten fraction. Therefore, to obtain information regarding this question and to isolate flour quality further, the technique of fractionating and reconstituting was applied so that the fat extracted with ethanol and petroleum ether from the gluten of one variety was replaced by the similarly extracted fat of the other in the reconstituted doughs. The results are shown in Figure 2.

The top row of Figure 2 shows that the volumes and inside characteristics for the three reconstituted Chiefkan loaves (Nos. 6, 7, and 8) containing the fatty material extracted from Kharkof gluten are equal to those for the Chiefkan check (Loaf 10), within the limits of experimental error. Likewise when the fat extracted from Chiefkan gluten was substituted for the Kharkof fat in the reconstituted Kharkof loaves (Nos. 3, 4, and 5) the loaf volumes and inside characteristics were equal to those of the check (Loaf 9).

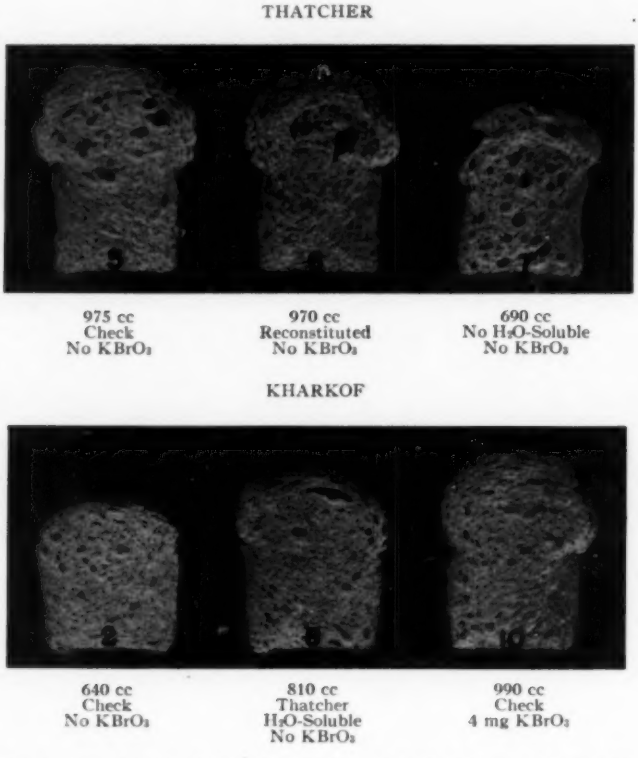


Fig. 3. Loaf volumes and inside characteristics of loaves obtained in experiments with the water-soluble fraction of Thatcher.

Thus neither the ethanol, the petroleum ether, nor the ethanol and petroleum ether extracts from the Kharkof and Chiefkan gluten fractions account for the difference in baking quality of the two varieties. These results, when considered with those associating quality with the gluten fraction, indicate that the extracted glutens contain the material responsible for their quality difference. A further study involving the purification of the gluten proteins is under way.

Importance and Function of Water-Soluble Fraction

The importance and function of the water-soluble fraction were also considered in a preliminary study. Thatcher was included, together with Kharkof and Chiefkan, in order to have represented the extremes in baking potentialities and in physical and chemical properties, such as bromate requirement, dough-handling properties, and mixing time. Flours of the three varieties were separated into starch, gluten, and water-soluble fractions as before and fractions of each were recombined to produce doughs with and without the water-soluble

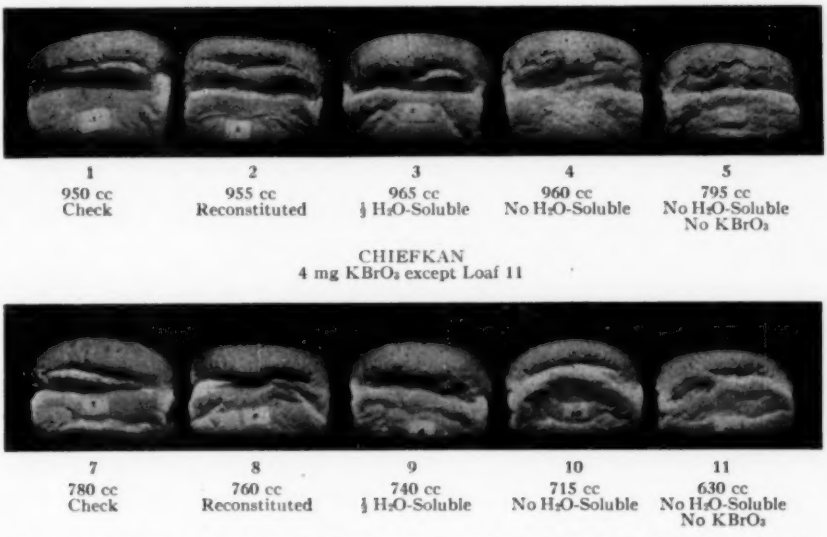


Fig. 4. Loaf volumes and outside characteristics of loaves from reconstituted Kharkof and Chiefkan doughs containing different amounts of their water-soluble fractions.

fractions. Also Chiefkan and Kharkof were reconstituted with half the water-soluble fractions. In one experiment water-soluble material from Thatcher was substituted for that of Kharkof. In certain cases bakes were made with and without bromate. The results are shown in Figures 3 and 4.

The reconstituted Thatcher containing no water-soluble material (Fig. 3, Loaf 7) gave a volume of 690 cc, which was 285 cc less than that for the Thatcher check. In other words, recombining the Thatcher starch and gluten alone gave a reconstituted dough far inferior to the original. A somewhat similar result was secured with Chiefkan (Loaves 7 to 10, Fig. 4), but the reduction in volume caused by omission of the water-soluble material was much less. Omission

of the water-soluble material in Kharkof (Loaves 1 to 4, Fig. 4) caused no reduction in volume whatever. These data suggest that the water-soluble material plays a much more important role in Thatcher than it does in the winter varieties Kharkof and Chiefkan.

The reconstituted Thatcher dough (Loaf 7, Fig. 3) was quite normal when mixed, but during fermentation became progressively more bucky. At the time of panning it behaved as though it had been heavily overbromated. The inside appearance of the loaf testified to the tightness of the dough and this tightness is further indicated by the proof heights given in Table III, together with those of Kharkof

TABLE III

EFFECT OF WATER-SOLUBLE FRACTION ON PROOF HEIGHTS OF DOUGHS PROOFED FOR 55 MINUTES

Treatment	Proof height		
	Kharkof	Chiefkan	Thatcher
	cm	*cm*	*cm*
Check	7.0	6.9	7.2
Reconstituted with normal amount water-soluble material	7.0	6.8	7.1
Reconstituted with ½ normal amount water-soluble material	7.1	6.6	—
Reconstituted with no water-soluble material	6.8	6.4	6.2

and Chiefkan. The external appearance of the Chiefkan without the water-soluble fraction (Loaf 10, Fig. 4) indicated a tight dough such as is usually obtained by employing too much bromate. In this case also the observed tightness is supported by the proof heights, which indicate ease of expansion. The proof-height data and the dough-handling properties suggest that the water-soluble fraction functions as a protein-softening or conditioning material.

Loaves 2 and 10 of Figure 3 represent Kharkof without bromate and with 4 mg of $KBrO_3$, respectively. Loaf 8, from a reconstituted Kharkof dough containing the water-soluble fraction from Thatcher and no bromate, is typical of the bread obtained from Kharkof dough containing about 1½ mg of bromate. This suggests that the water-soluble fraction from Thatcher had greater oxidizing properties than similar material from Kharkof. The loaf-volume and inside characteristics of Thatcher with no bromate (Loaf 5, Fig. 3) when compared to those of Kharkof with no bromate (Loaf 2, Fig. 3) and with 4 mg of bromate (Loaf 10, Fig. 3) illustrate the marked difference in bromate requirement of spring and winter varieties.

Loaf 5 of Figure 4 was obtained from a reconstituted Kharkof dough containing no water-soluble material and no bromate. Its

volume (795 cc) is 155 cc larger than the Kharkof check in Figure 3 (Loaf 2), which also was baked without bromate and is typical of the bread usually obtained from Kharkof with 1 to 1½ mg of bromate. In a similar manner Chiefkan with no water-soluble fraction and no bromate (Loaf 11, Fig. 4) produced a loaf 30 cc larger than that obtained with the original Chiefkan flour and no bromate (not shown). These results suggest that the water-soluble material has a reducing action. They might also be interpreted as indicating that, for those varieties with larger bromate requirements, such as Kharkof, and to a lesser degree Chiefkan, there is contained in the water-soluble fraction much more of that reducing constituent required for a proper conditioning of the dough than is contained in the water-soluble fraction obtained from such varieties as Thatcher, which is characterized by a small bromate requirement. Whether this is a general relation or is peculiar to the three varieties studied herein must await further study.

Data obtained by Finney and Barmore [4] and by Barmore, Finney, and McCluggage (1941), show that within a variety the bromate requirement increases as the protein content rises. To determine whether this increase in bromate requirement is due to the water-soluble fraction, some preliminary experiments were carried out in which exceptionally high protein levels were obtained by fortifying the original flour with gluten. For this study, to each of three flours from Kharkof, Chiefkan, and Thatcher there were added 3, 6, and 9 g of their corresponding glutens. The amount of flour fortified in every case was such as to give 100 g of the fortified product. Each of the three Kharkof gluten-fortified flours was baked with 4 mg of $KBrO_3$, and with additional amounts of the Kharkof water-soluble fraction in proportion to the quantity of gluten protein that was added. Thus for 100 g of flour yielding 20 g of dry gluten and 30 g of water-soluble material, there was added for each 3 g of gluten 3/20 of 30 or 4.5 g of the water-soluble fraction. Three identical gluten-fortified flours were also baked with the added water-soluble fraction but with 5 mg of bromate for the first addition of gluten and 6 mg for the second and third additions. The three gluten-fortified Kharkof flours were baked a third time without added water-soluble material and with only 4 mg of $KBrO_3$.

Chiefkan and Thatcher were treated in a similar manner, the same increments of bromate being used for Chiefkan as were used for Kharkof. For Thatcher, however, 2 mg of bromate was used where 4 mg was used for Kharkof, and 2½ mg was used for the first and second

[4] Presented at the 25th and 27th Annual Meetings of the A. A. C. C., 1939 and 1941, Kansas City and Omaha, respectively.

gluten additions and 3 mg for the third. One lot of flour of each variety was included in which the protein level was reduced by adding starch. The results are shown in Figure 5.

In all cases the highest loaf volumes were obtained when additional bromate and water-soluble material were added to the gluten-fortified flours. Additional water-soluble material without additional bromate (at the higher protein levels) produced almost identical results with

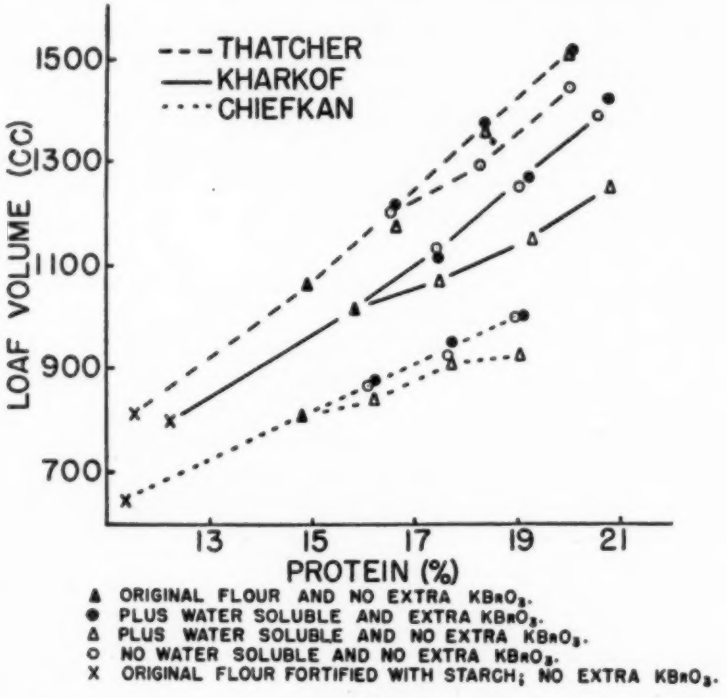


Fig. 5. Results of experiments with the water-soluble fraction and $KBrO_3$ at the higher protein levels obtained by adding gluten to the original flour.

Thatcher, but for Kharkof and Chiefkan the loaf volumes were materially lower. On the contrary, when neither bromate nor water-soluble materials were added there was no reduction in loaf volume with Kharkof, practically none with Chiefkan, but a material reduction with Thatcher. The results are in accord with those presented above in suggesting that the water-soluble fractions contain materials associated with the bromate requirement of the flour.

Loaf Volume at Abnormal Protein Levels

Loaf-volume and protein-content data obtained in this laboratory with numerous hard winter and hard spring wheat flours have given

regression lines which, when extrapolated, cross at a protein content of 7% to 8%. On this basis the illogical conclusion could be drawn that varieties considered of poor quality at the medium and high protein levels are superior in quality at low protein levels. To clarify this question the technique of fractionating and reconstituting flour was applied to obtain flours both above and below the protein range usually encountered. Another objective was to determine whether the relation is completely linear or slightly curvilinear over the normal protein range.

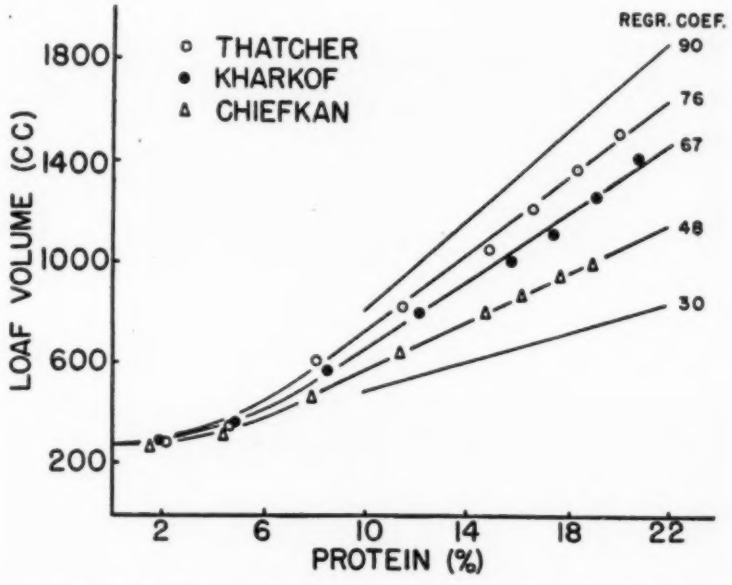


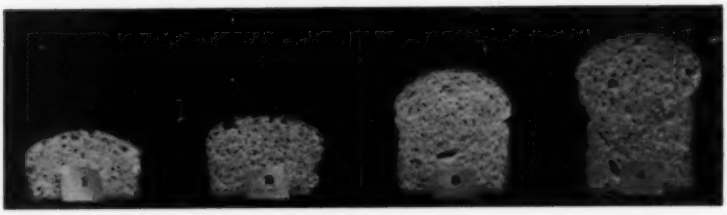
Fig. 6. The relation of loaf volume and protein content using fractionation and reconstitution methods in making up the flour-doughs.

An even more important application of this technique was designed to find the extent to which the regression coefficient, or loaf-volume correction factor, is related to the protein content and loaf volume.

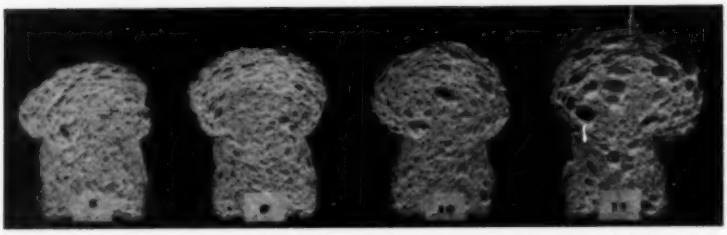
In this study the separate fractions of starch, protein, and water-soluble material from each of the three varieties previously described were added back to the flour from which they were obtained in such a manner as to produce flours varying in protein content from about 1.5% to 21%. The results for this study are shown graphically in Figure 6. Figure 7 shows the inside characteristics of the loaves representing the Kharkof and Chiefkan series.

Figure 6 shows that the lines do not cross in the low-protein levels but instead become definitely curvilinear below about 7% and meet at 0% protein and about 275 cc loaf volume.

KHARKOF

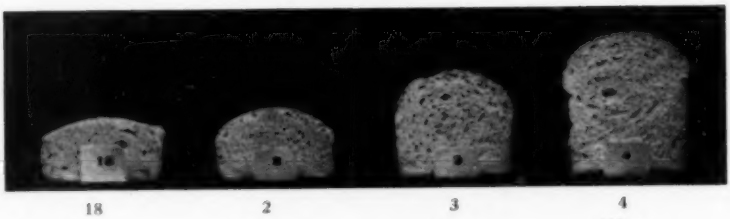


1	2	3	4
290 cc 1.87%	363 cc 4.91%	575 cc 8.56%	803 cc 12.18%

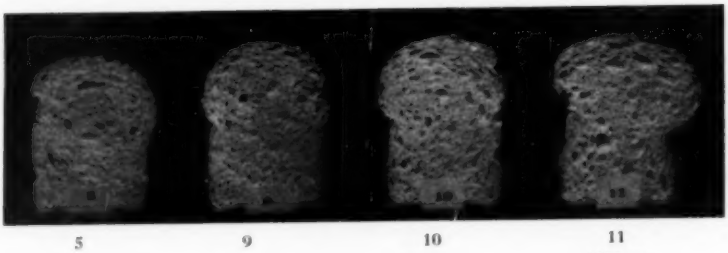


5	9	10	11
990 cc 15.80%	1120 cc 17.43%	1265 cc 19.16%	1420 cc 20.73%

CHIEFKAN



18	2	3	4
275 cc 1.56%	310 cc 4.45%	480 cc 7.92%	640 cc 11.35%



5	9	10	11
808 cc 14.80%	875 cc 16.20%	950 cc 17.71%	1000 cc 19.06%

Fig. 7. Loaf volumes, inside characteristics, and flour protein contents for loaves representing a Chiefkan and a Kharkof protein series including sub-normal protein levels. The flour-doughs were obtained by fractionation and reconstitution methods.

The slopes of the regression lines between the limits of about 8% and 20% protein are approximately 67, 48, and 76 for Kharkof, Chiefkan, and Thatcher, respectively. The lines with slopes of 90 and 30 have also been drawn in to complete the picture for later reference. Within these limits the relation between protein content and loaf volume is practically linear. Thus the regression coefficient, or correction factor for protein content, can be applied over the range of approximately 8% to at least 20% protein. Also, the regression coefficient appears to be a function of the loaf volume at any arbitrary protein content within this range. Therefore, one can determine the factor for correcting the loaf volume of any sample or variety to a constant protein basis by determining its protein content and loaf volume and then locating this point on a graph such as Figure 6.

Figure 7 shows that for both Kharkof and Chiefkan a protein content of about 8% (Loaf 3 in each case) still produces loaves with quite normal inside and outside characteristics so clearly lacking at the lower protein levels. During fermentation the doughs below 8% protein appeared to be incapable of retaining the gas produced and this was evident from the porous appearance of Loaves 1 and 2. Apparently a continuous phase of protein is absent when the flour contains much less than 8% protein.

Summary and Conclusions

Flours of two varieties of winter wheat, Chiefkan and Kharkof, and of one spring wheat, Thatcher, representing a wide range in quality characteristics, were fractionated into starch, gluten, and water-soluble fractions which were then recombined in the original and in different proportions and then baked into bread along with the original nonfractionated flours. Gluten fats were also extracted with ethanol, petroleum ether, and with ethanol followed by petroleum ether. Also the various fractions of some varieties were interchanged before baking.

In other experiments the flour protein content of the three varieties was increased by adding varying quantities of their own gluten and in some cases reduced by adding starch. These various techniques permitted a determination of the factors responsible for differences in bread quality and also a more accurate study of the relation between protein content and loaf volume. The results obtained appear to justify the following conclusions:

By the techniques employed it is possible to fractionate flours as above described, recombine them in their original proportions, and secure bread equal to that made with the original or nonfractionated flour within the limits of random error. This indicates that the fractions were not significantly altered in making the separation.

The recognized differences in bread quality of Chiefkan and Kharkof were entirely accounted for by differences in their gluten fractions.

The fat extracted from Kharkof and Chiefkan gluten did not account for any of the difference in their baking quality. Therefore the glutens extracted with fat solvents still contained the material responsible for their quality difference.

Omission of the water-soluble fraction in reconstituted doughs of Kharkof flour resulted in no reduction in loaf volume or bread quality. Omission of this same material in Chiefkan dough resulted in a significant loaf volume reduction. For Thatcher this volume reduction was extremely large. It appears that the water-soluble fractions contain protein-softening or conditioning materials which vary in amount or composition for different varieties.

The relation between protein content and loaf volume was found to be substantially linear between the limits of 7% or 8% to at least 20% protein, provided adequate amounts of bromate were used, especially at the higher protein levels. Therefore the regression coefficient, or loaf volume correction factor for protein content, can be applied within these limits. Below 7% protein the relation was definitely curvilinear, all curves meeting at 0% protein and about 275-cc loaf volume.

The regression of loaf volume on protein content is different for different varieties and appears to be a function of the loaf volume that may be produced by a variety at any arbitrary protein level within the range of linearity. Therefore, one can determine the factor for correcting the loaf volume of a given sample to a constant protein basis from a knowledge of its protein content and loaf volume, thus eliminating the need of loaf volume data at several protein levels of the variety or sample in question.

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BOOK REVIEW

Food Manufacturing. By Saul Blumenthal. 664 pages. Chemical Publishing Co., Brooklyn, N. Y. Price $7.50.

This book contains a great deal of practical and useful information, being essentially a compilation of recipes and formulas pertaining to almost every known type of commercial food manufacture and processing.

The author has essayed to cover so much territory that his treatment of individual topics is sometimes sketchy and incomplete. For example, on page 2 a table giving the relationship between steam pressure and temperature—up to 100 lbs. pressure—is shown, but is not related to the processing of any commodity. Similarly on page 3, under Desiccation (which is misspelled) no mention is made of final moisture content, times and temperatures to be recommended, or the necessity for scalding vegetables as a preliminary step.

On page 472 the object of blanching vegetables intended for freezing is not mentioned nor is there any statement of the proper storage temperature (important from the quality standpoint) for frozen fruits or vegetables. The author implies that frozen vegetables are a luxury article, with only a limited market. This is not entirely compatible with the Army's announcement of its intention to purchase a minimum of 71,000,000 pounds in 1943.

On page 9, it is stated that fruit and berries are free from mold after washing. This is an unfortunate statement.

There is a short chapter on composition, analyses and tests of the principal foodstuffs and of various ingredients and flavoring materials that are of interest to the commercial food processor.

J. A. Berry, Western Regional Research Laboratory,
Bureau of Agricultural and Industrial Chemistry,
Agricultural Research Administration,
U. S. Department of Agriculture

SUGGESTIONS TO AUTHORS

General Arrangement of Manuscript: Send one copy, double-spaced throughout except for footnotes, with about inch-width margins. Place each table on a separate sheet. Throughout the text insert footnotes, single spaced, with a solid rule above and below, just below the lines in which the reference numbers occur. Indicate appropriate places for figures and tables by typing "(Table I)" or "(Table II)" etc., or "(Fig. 1)" or "(Fig. 2)" etc., in the middle of the line. Submit the illustrations as separate items with numbers and authors' names on the back, reduced to convenient size. Make a separate list of legends for illustrations.

Tables: For tabulations use the type of arrangement found in previous numbers of CEREAL CHEMISTRY. Suggestions that may be useful in simplifying and reducing the size of tables are as follows: Omit columns if the data represent simple calculations from data in other columns. Omit columns that contain only a few data. Omit data that are of value only to the author. Omit columns that do not show significant variations. Limit tables to approximately 8 columns so that they can be placed horizontally on the page. If the tabulation is small and no correlation is shown, use a "leader" table, one without number and title.

Line Drawings and Photographs: These should have serial figure numbers and legends. Submit copy on 8½ × 11-inch paper or smaller. Large graphs may be reduced by the photostatic or photographic process.

The author should if possible have all line drawings made by a competent draftsman. Use the horizontal scale for independent and the vertical scale for dependent variables. It is usually recommended that omissions in the scales be shown by breaking. Symbols such as circles and triangles cannot be set in type and consequently must be explained in a legend on the drawing. The lettering should be large enough so that after reduction it will be from 1/16 to 1/8 inch high. Use a fine pen for coordinates and a heavier pen for the curves and borders. Avoid waste space; make the graph neat and well balanced. Enclose all sides with border lines.

Glossy photographic prints are best and a width of not less than 5 inches is convenient. The 5-inch width is a standard size and is near to page width. Keep in mind the page width (4¼ inches) in arranging objects to be photographed. Use the vertical page dimension (7 inches) only in cases of necessity.

Text: The title should be specific and long enough to name the factors involved in the investigation. Avoid unnecessary words.

The introduction and review of literature should deal only with those matters *most closely* related to the investigation. To avoid an extremely long review of literature the following methods of elimination are suggested: (1) Cite a competent, recent review and let it serve in place of a similar review. (2) Consider the literature reviewed in the order of relationship to the problem at hand and eliminate the less closely related references.

Methods of statistical analysis do not require explanation. Treat them as you would other standard procedures; that is, interpret the results but do not present the method.

Edit sentences with the object of reducing the number of words. In sentence construction select strong specific subjects, use active verbs as much as possible, and delete all unnecessary modifiers. The language of technical papers is commonly a subject for severe criticism.

Use center headings for the larger sections and divide long sections by means of sideheads. Use only these two types of subdivisions.

Always include a summary, consisting of brief unnumbered paragraphs. Place the acknowledgments between the summary and the list of literature cited. Follow the style of previous issues in citing literature and preparing the list of literature cited. Acknowledge the use of unpublished matter in a footnote or in the text.

Abbreviations: For publications use the abbreviations found in the list of periodicals abstracted in CHEMICAL ABSTRACTS (1936). Do not use periods following abbreviations. Use *ml* instead of *cc*, except for loaf volumes. Abbreviate freely in tables, and if the abbreviations are not standard show their meaning in a footnote.

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Recent research shows that copper and iron, even in minute traces, are "bad actors." In the manufacture of shortening, they are eliminated to increase rancidity resistance of fats and oils. They affect stability of dairy, bakery, and meat products —and practically any food that contains traces of fats or volatile flavors easily oxidized.

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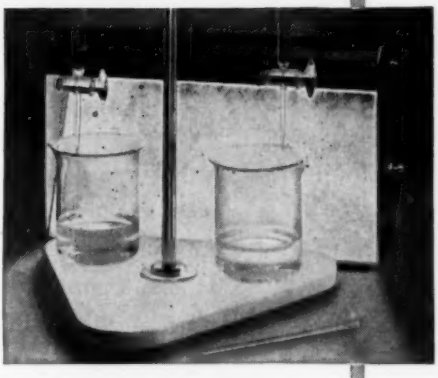
A *parabolic* reflector directs light against a flat, white, acid resistant vitreous enameled inclined background—eliminating direct illumination and so preventing glare and undesirable illumination intensities.

The outer surface of the lamp is finished in a dark blue vitreous enamel. Fitted with snap-switch, connecting cord and plug. Length, 12½ inches; height, 8 inches; depth, 4½ inches.



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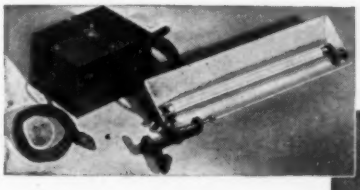

The lamp can be attached to all makes and styles of balances. Mounted at the front of a balance case, the *parabolic* reflector directs all light through the front door over the entire working area of a balance to uniformly illuminate beam, chain scale, pointer index and pans. Objectionable shadows and poorly lighted areas are entirely eliminated.

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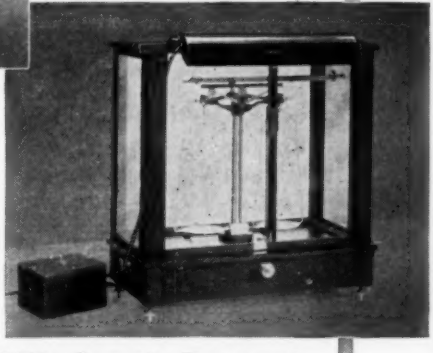

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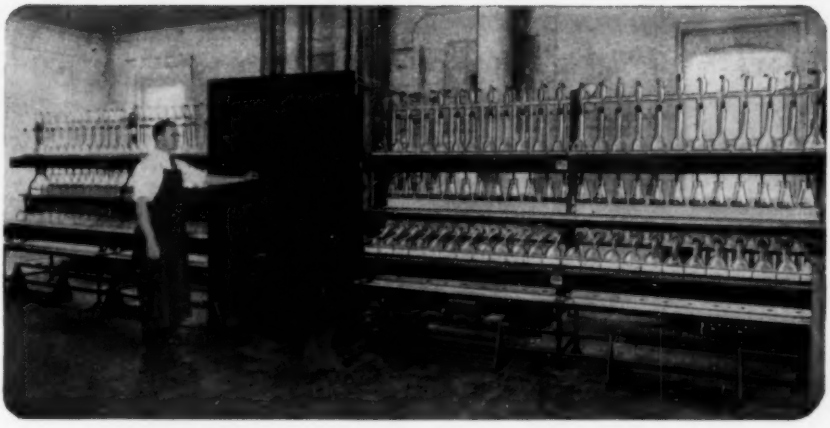

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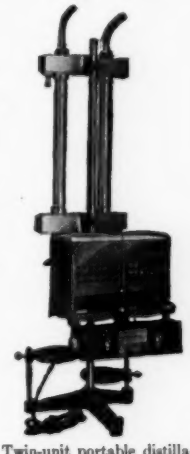

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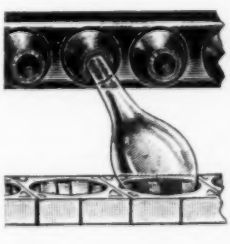

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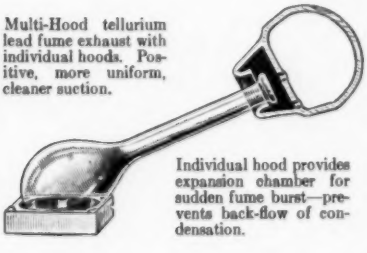

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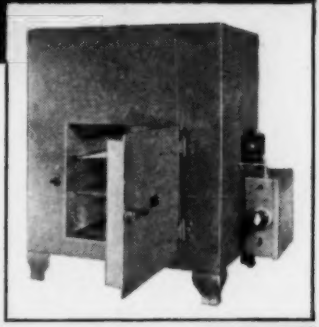

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